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PEI Associates, Inc., Cincinnati, OH

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16. ABSTRACT

This inspection guide was written and organized for use by State and local enforcement field inspectors. The purpose of this guide is to aid in the development of uniform evaluation procedures to determine compliance with NSPS requirements for nitric acid plants. The manual describes the operating principles for nitric acid plants and control techniques that may be used to determine compliance with regulatory requirements.

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DISCLAIMER

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SECTION 1

INTRODUCTION

New Source Performance Standards (NSPS) have been promulgated by the U.S. Environmental Protection Agency (EPA) for nitric acid (HNO_3) plants producing weak nitric acid (30 to 70 percent strength) as authorized under Section 111 of the Clean Air Act (42 U.S.C. Section 1857 et seq.), as amended. Under Section 111, the Administrator of EPA is authorized to develop and promulgate standards of performance for new stationary sources that are considered to contribute significantly to air pollution. The nitric acid standards of performance that were promulgated December 23, 1971, apply to all nitric acid plants constructed or modified after August 17, 1971.

Each State agency may develop its own enforcement program for NSPS sources within its jurisdiction. If the program is determined to be adequate, the State agency may be delegated authority to oversee enforcement of the NSPS. This authority would be granted over all new and modified sources subject to a particular NSPS regulation, provided the emission limitations adopted at the State level for the NSPS sources are at least as stringent as the Federal NSPS standards.

Effective implementation and enforcement of the NSPS program depends primarily on the inspection program used to ensure compliance with the applicable emission limitations. For nitric acid plants, this requires a mix of the recordkeeping requirements and periodic field surveillance.

The purpose of this manual is to aid in the development of uniform evaluation procedures to determine compliance with the NSPS requirements for nitric acid plants. This manual discusses the operating principles for nitric acid plants and the control techniques that may be employed to meet emission limitations and describes inspection techniques that may be used to determine compliance with the regulatory requirements.

This manual is divided into 7 sections. Section 1 is the introduction. The other six sections are summarized as follows:

- Section 2 - This section briefly outlines the requirements of the NSPS for nitric acid plants and those of the State Implementation Plans (SIP's). It is designed to aid and familiarize enforcement personnel with the regulations applicable to nitric acid plants.
- Section 3 - This section is intended to help familiarize personnel with the processes and instrumentation involved in the production of nitric acid, the control equipment that may be applied to reduce emissions, and the level of emissions or degree of control that may be expected from each type of control equipment.
- Section 4 - This section discusses some of the effects of startup, shutdown, and malfunctions on the emissions from nitric acid plants and some of the steps that may be taken to reduce or minimize their impact.
- Section 5 - This section discusses the operating parameters that should be observed during the performance test for demonstration of compliance. In addition, this section discusses the continuous emission monitoring testing requirements.
- Section 6 - This section discusses recordkeeping requirements, data required in these records, emission monitoring techniques, and methods for evaluating performance and excess emission reports.
- Section 7 - This section discusses inspection techniques that may be used by enforcement personnel to determine compliance with applicable limitations during routine field surveillance and followup inspections.

This manual also contains three appendices:

Appendix A: Nitric Acid NSPS Regulations

Appendix B: EPA Methods 1, 2, 3, and 7

Appendix C: Glossary of Terms.

1.1 SIP REQUIREMENTS

Existing nitric acid production facilities not subject to NSPS are subject to the emission limitations contained in the SIP's. Most States limit the emissions from nitric acid plants through a general process weight rate regulation. The following States, however, limit the emissions as a result of regulations that are specific for nitric acid plants:

Alabama
Arizona
Connecticut
Georgia
Kentucky
Louisiana
Maryland
Minnesota
Nebraska
North Dakota
Pennsylvania
South Dakota
Tennessee
Texas
Virginia
Washington
Wisconsin
Wyoming

Table 1 summarizes the SIP limits for the States that have specific requirements for nitric acid plants. The nitrogen dioxide (NO_2) limits from existing nitric acid plants generally range from 3.0 to 5.5 lb/ton of 100 percent acid produced.

TABLE 1. SUMMARY OF SIP REQUIREMENTS SPECIFIC TO NITRIC ACID PLANTS

State	Pollutant	Limit, lb/ton of 100% acid
Alabama	NO_2	5.5
Arizona	NO_2	3.0[a,b,c] 5.5[b,c,d]
Connecticut	NO_2	5.5
Georgia	NO_2	25.0[e,f] 3.0[f,g]
Kentucky	NO_2	3.0[f,h,i,j] 5.8[i,k]
Louisiana	NO_2	6.5
Maryland	NO_2	3.0
Minnesota	NO_2	3.0[c,l] 5.5[c,m]
Nebraska	NO_2	5.5
North Dakota	NO_2	3.0[f,i,j]
Pennsylvania	NO_2	5.5
South Dakota	NO_2	-[j,k]
Tennessee	NO_2	3.0[c,n] 5.5[c,o]
Texas	NO_2	600[p]
Virginia	NO_2	5.5
Washington	NO_2	5.5
Wisconsin	NO_2	3.0[b]
Wyoming	NO_2	3.0[b,c,h] 5.5[b,c,k]

[a] Plant under construction or in production on or after August 20, 1973.

[b] Weak acid (30-70% in strength).

[c] Maximum 2 hour average.

[d] Plants under construction or in production before August 20, 1973.

[e] Plants constructed before January 1, 1972.

[f] Must be equipped with a continuous NO_x monitor and recorder or an alternate system approved by the Director.

[g] Plants constructed after January 1, 1972.

[h] Plants under construction or in production before August 17, 1971.

[i] Opacity not to exceed 10 percent.

[j] Owner or operator must record the daily production rates and hours of operation.

[k] Plants under construction or in production before August 17, 1971.

[l] Plants under construction or in production on or after February 1, 1972.

[m] Plants under construction or in production before February 1, 1972.

[n] Plants under construction or in production on or after April 3, 1972.

[o] Plants under construction or in production before April 3, 1972.

[p] Parts per million by volume.

SECTION 2

NSPS REQUIREMENTS AND SIP EMISSION LIMITATIONS

2.1 NSPS REQUIREMENTS

Standards of Performance for new nitric acid plants producing weak nitric acid were published and promulgated in the Federal Register on December 23, 1971. The regulation (see Appendix A) applies to all plants producing 30 to 70 percent strength nitric acid that are constructed or modified after August 17, 1971. Designated Subpart G of the NSPS, the regulations are summarized below. In addition to the requirements of Subpart G, nitric acid plants subject to NSPS are also subject to the monitoring and recordkeeping provisions of Subpart A.

The current emission standards were reviewed in 1979 and 1984 by EPA to determine if any changes in the current emission limitations were warranted. No revisions to Subpart G and the emission standards are proposed at this time.

2.1.1 Emission Standards

The allowable emissions limits for nitrogen oxides and opacity are stated in Section 60.72. Emissions of nitrogen oxides are limited to less than 1.5 kg (expressed as NO_2) per metric ton of nitric acid produced (3.0 lb/ton). Nitric acid production is expressed as 100 percent nitric acid. Opacity is limited to 10 percent or less.

2.1.2 Performance Testing

Determination of compliance is made according to performance tests conducted according to Section 60.8 of the NSPS. This section requires the plant operator to provide a written report of the tests that must be conducted at conditions representative of the normal operating conditions. Testing for nitrogen oxides is conducted using Method 7 and the criteria of Methods 1 and 2.

Performance tests are required to be conducted within 60 days of achieving maximum production rate, but not longer than 180 days after initial startup. The EPA must receive at least 30 days notice prior to all performance tests. In addition, the plant owner or operator is responsible for providing the following performance testing facilities:

1. Sample ports adequate for applicable test methods,
2. Safe sampling platform(s),
3. Safe access to sampling platforms, and
4. Utilities for sampling and testing equipment.

Tests in addition to the initial performance test may be required from time-to-time by the Administrator as authorized under Section 114 of the Clean Air Act.

2.1.3 Stack and Process Monitoring

Nitric acid production facilities are required to install, operate, and maintain a continuous emission monitor for the measurement of nitrogen oxides (NO_x). Because the units of the emission standard are expressed as NO_2, the emission monitor should display the output as NO_2. Requirements for these continuous monitoring systems are outlined in Section 60.73, Section 60.13, and Appendix B of 40 CFR 60 - Performance Specification 2.

The monitoring system is required under Section 60.13 to meet the requirements of Performance Specification 2. This performance evaluation is required to be conducted within 30 days of any performance test required under Section 60.8 and at any other time it is deemed necessary by the Administrator.

In addition to operating and maintaining the continuous monitor, the plant operator must perform zero and span checks on the monitor at least once every 24 hours. The plant operator may zero and span the instruments at more frequent intervals, if desired.

The plant owner or operator must record daily production rates and hours of operation. A conversion factor established during the performance test (Section 60.8) or performance evaluation (Section 60.13) to convert from ppm NO_2 to units of the standard shall be used to determine excess emissions. These excess emissions are calculated as the arithmetic mean of any three contiguous 1-h periods as measured by the continuous monitoring system exceeding the standard in Section 60.72(a) (1.5 kg/metric ton).

2.1.4 Recordkeeping and Reporting

The recordkeeping and reporting requirements for nitric acid plants subject to NSPS are outlined in Section 60.7. Enforcement personnel should be particularly aware of the requirements of Section 60.7(b) and (c). Section 60.7(b) requires the plant owner or operator to maintain records of any startup, shutdown, or malfunctions that occur at an affected facility, any malfunction of control equipment, or any periods in which the continuous monitor is inoperative. In addition, Section 60.7(c) requires the plant owner or operator to submit a quarterly excess emission report postmarked by the 30th day following the end of each calendar quarter. The excess emission report must include the identification of any periods of excess emissions, including excess emissions that occur during periods of startup, shutdown, and malfunctions. The report must also identify the nature and cause of the malfunction and the corrective action taken with respect to the malfunction. In addition, the excess emission report must include the date(s) and time(s) of any periods when the continuous monitor was inoperative (except for daily zero and span calibration checks) and the nature of repairs or adjustments necessary to restore monitor operation.

The plant owner or operator must maintain a record, in a manner suitable for inspection, of all monitoring data, maintenance records, performance tests, continuous monitor performance evaluations, and excess emission reports for a period of two years. These requirements are outlined in Section 60.7(d).

SECTION 3

PROCESS DESCRIPTION, ATMOSPHERIC EMISSIONS, AND EMISSION CONTROL METHODS

3.1 PROCESS DESCRIPTION

Presently all weak nitric acid is manufactured by the ammonia oxidation process (AOP). In general, this process involves the oxidation of ammonia by air (oxygen) with subsequent cooling and water absorption of the reaction gases to produce nitric acid.[1,2] A majority of AOP plants are located at or near ammonia plants, and the product acid is used in the production of nitrates. Thus, ammonium nitrate plants are usually [illegible]ted in the same production complex with ammonia and nitric acid plants.

Earlier processes, based on the reaction of sulfuric acid on saltpeter or the passing of air through an electric arc, have been replaced and anticipated breakthroughs for the direct combination of nitrogen and oxygen in nuclear reactors has not taken place. Also with the new generation of ammonia plants cutting the cost of ammonia in half, it appears unlikely that other raw materials for nitric acid would become competitive in the near future and therefore, the ammonia oxidation process will remain the main source of weak nitric acid production.

3.1.1 Basic Process Description of AOP

Despite many variations in operating details among the plants producing nitric acid, three basic steps are common to all: 1) oxidation of ammonia to nitric oxide, followed by 2) oxidation of nitric oxide to NO_2, and finally 3) absorption of NO_2 in water to produce nitric acid with the release of additional nitric oxide.

The essential equipment and flow sequence in AOP are illustrated in Figure 1.[3] Ammonia vapor is mixed with preheated air and passed over a catalyst, where the ammonia is oxidized to nitrogen oxide. The hot reaction gases are cooled in waste heat boilers (for heat recovery and steam generation) and heat exchangers, permitting the nitric oxide to be oxidized to NO_2 by the

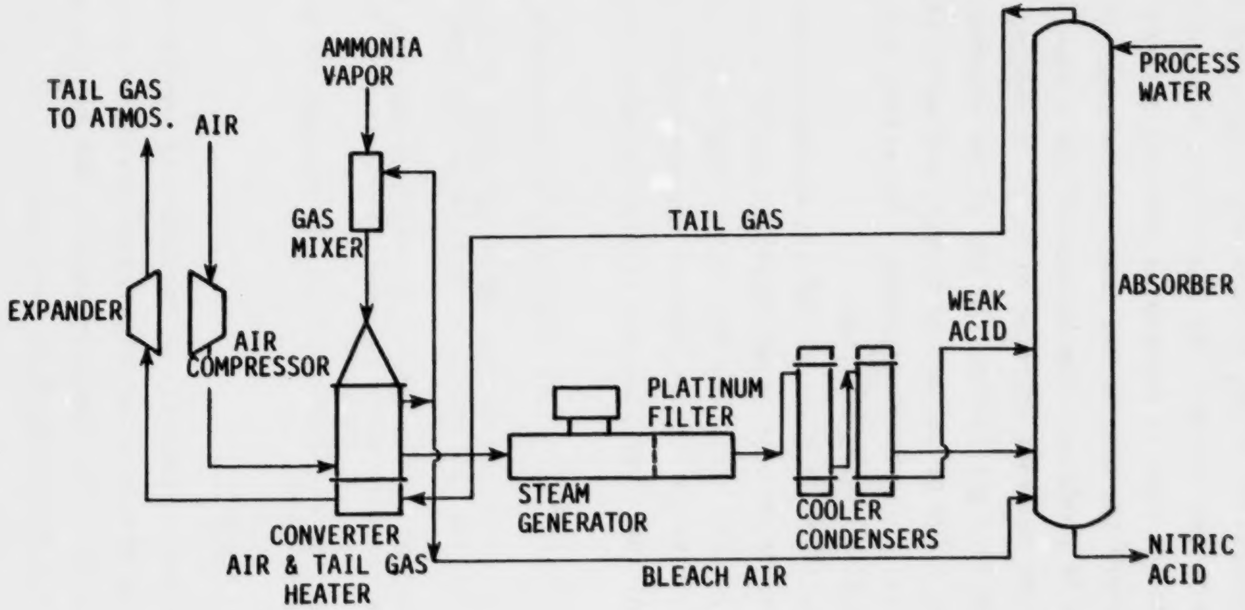

TAIL GAS
TO ATMOS.
AIR
AMMONIA
VAPOR
GAS
MIXER
EXPANDER
AIR
COMPRESSOR
CONVERTER
AIR & TAIL GAS
HEATER
TAIL GAS
STEAM
GENERATOR
PLATINUM
FILTER
COOLER
CONDENSERS
WEAK
ACID
BLEACH AIR
PROCESS
WATER
ABSORBER
NITRIC
ACID

Figure 1. Basic ammonia oxidation process.[3]

oxygen remaining in the process gas stream. On cooling, water condenses and some NO_2 dissolves to form weak nitric acid. The process gas then flows to an absorber where the nitric acid is formed by absorption of NO_2. Nitric oxide is released in the absorption of NO_2 to form nitric acid and the nitric oxide passes through the absorber column. The weak nitric acid produced in the cooling and NO_2 formation step is introduced into the absorber at the point in the vertical tower where the acid in the tower is the same concentration as the nitric acid that is produced. The balance of the oxygen (bleach air) required to oxidize the released nitrogen oxide is introduced into the bottom of the absorber. The tail gas leaving the top of the absorber contains residual nitrogen oxides, a low percentage of oxygen, and water vapor with the balance of the material being inert nitrogen. The nitric acid produced is withdrawn from the bottom of the absorber.

The chemical reactions involved in AOP are exothermic and the heat produced is used to generate process steam superheat and/or to evaporate the ammonia and to heat process air. The tail gas is typically reheated and expanded through a turbine to recover part of the compression power. Many plants are self-sustaining after they are started by using auxiliary energy sources.

In summary, the essentials of an ammonia oxidation nitric acid plant are:

1. Converter or oxidation section where the ammonia vapor and air are mixed and reacted catalytically to oxidize the ammonia.
2. Cooler-condenser section where the nitrogen dioxide is produced by cooling the reaction gases and weak nitric acid is formed.
3. Absorber section where the cool NO_2 is absorbed in water to form the product nitric acid.

3.1.2 Basic Process Chemistry

Knowledge of the basic chemistry is necessary to understand the commercial plant designs where the pressure and temperature characteristics of AOP chemistry are most efficiently used. The chemistry used in AOP for the manufacture of nitric acid involves the following three reactions:

$$4\ NH_3 + 5\ O_2 \rightarrow 4\ NO + 6\ H_2O \qquad (1)$$

$$2\ NO + O_2 \rightarrow 2\ NO_2 \rightarrow N_2O_4 \qquad (2)$$

$$3\ NO_2 + H_2O \rightarrow 2\ HNO_3 + NO \qquad (3)$$

The first reaction (1) is a heterogeneous gas-phase catalytic reaction. The primary oxidation of ammonia to nitric oxide, with a platinum alloy catalyst, is carried out at approximately 1500° to $1750^\circ F$, with a very short contact time. This reaction has been extensively studied, but no completely satisfactory reaction mechanism has resulted. The reaction rate is very rapid with a 95 percent or more yield of nitric oxide under a fairly wide range of conditions. In addition to this main reaction, other side reactions are defined and discussed in Reference 1.

The results of earlier experiments (Figure 2) show the relationship between contact time (inverse of gas velocity) and catalyst temperature.[4] These data indicate that the conversion of ammonia to nitric oxide can be maximized for each gas velocity by the optimum catalyst temperature. This reaction is so rapid that the percent conversion ultimately depends on the rate at which ammonia and oxygen can be contacted with the catalyst surface.

Figure 3 shows the effect of pressure on the ammonia-oxidation reaction. Increased pressure reduces the maximum amount of conversion that can be obtained for a given temperature and contact time.

The second reaction (2) is a homogeneous, noncatalytic, gas-phase reaction between nitric oxide and oxygen to produce NO_2. This is not only a slow reaction, but it is also strongly dependent on temperature. This reaction is unique in that the reaction rate decreases with increasing temperature as shown in Figure 4. This reaction is also strongly dependent on pressure. Figure 5 shows that increasing pressure causes a higher percent oxidation of nitric oxide and also reduces the reaction time necessary to reach maximum conversion in the process.

The third reaction (3) is a heterogeneous equilibrium reaction and consists of the absorption of NO_2 in water to form nitric acid. The rate of this reaction is controlled by the following steps:

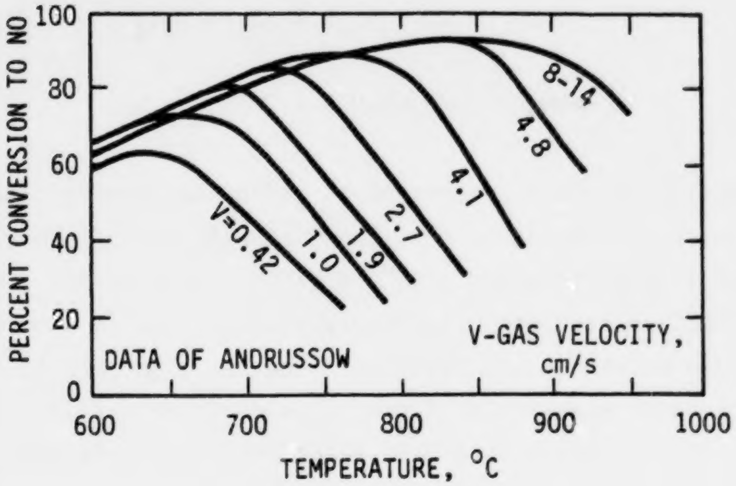


Figure 2. Conversion of ammonia to nitric oxide as a function of temperature and gas velocity.[5]

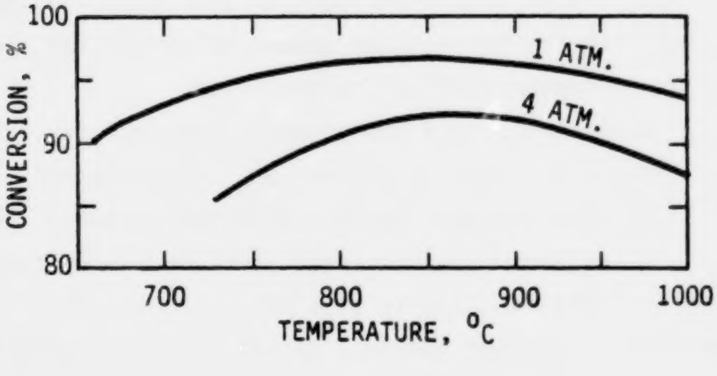
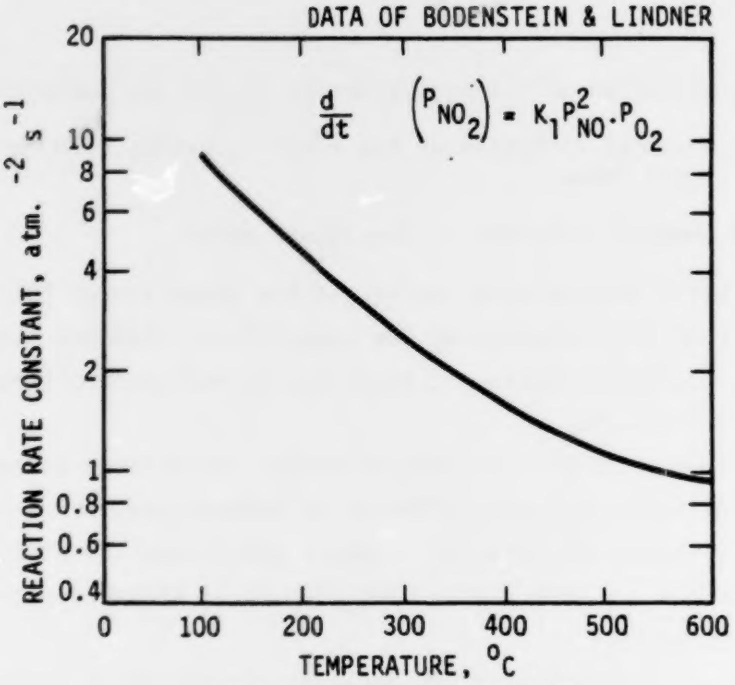


Figure 3. Effect of pressure on the oxidation of ammonia at different temperatures.[6]



RATE CONSTANT FOR REACTION

$2\ NO + O_2 \longrightarrow 2\ NO_2$

Figure 4. Rate constant.[7]

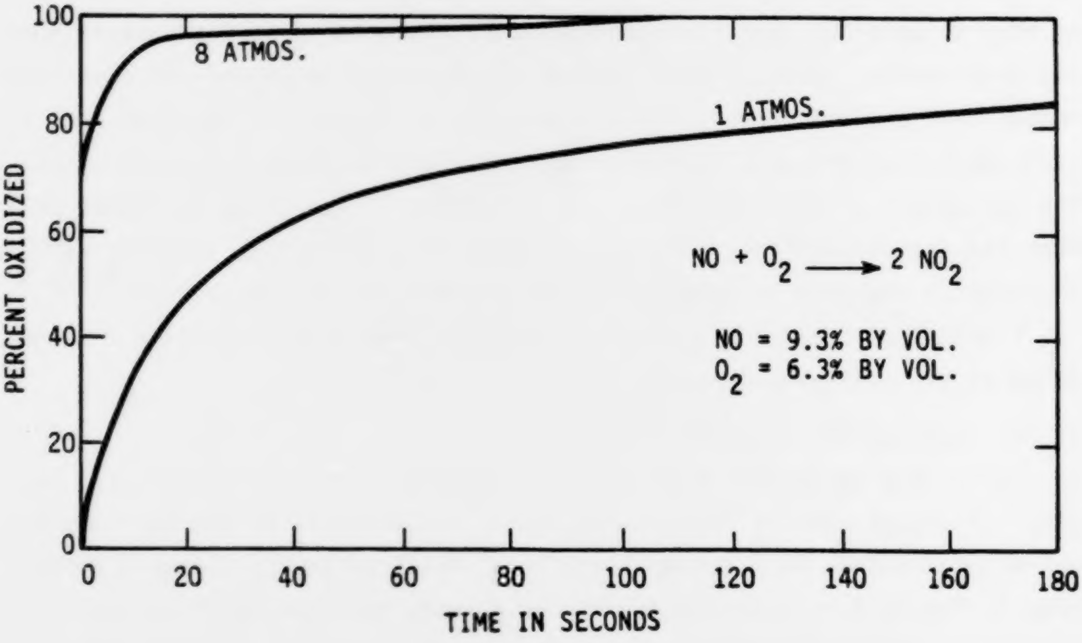


Figure 5. Rate of oxidation of ammonia converter products at 100°C.[1]

1. The oxidation of nitrogen oxide to NO_2 in the gas phase.
2. The physical diffusion of the reacting oxides from the gas phase to liquid phase.
3. The chemical reaction in the liquid phase.

Both steps 1 and 2 proceed more rapidly at low temperatures than high temperatures, while step 3 is retarded by low temperature. Because step 3 is inherently rapid even at low temperatures, it tends not to be a controlling factor in the formation of nitric acid.

The equilibrium conditions that determine the maximum strength of the nitric acid are not appreciably affected by temperature, but are favorably affected by increased pressure, which makes the production of higher acid strengths possible. Figure 6 shows the effects of elevated pressure on the overall absorption rate.

Although the efficiency of AOP declines slightly with increased operating pressure, the nitric oxide oxidation and the absorption reaction rates are greatly increased by increased pressure.

3.1.3 Commercial Processes for Weak Acid Production

Before the development of stainless iron and steel alloys in the 1920's, the only material suitable for absorption of nitric acid were stoneware and acid-proof brick. Because this limitation prevented operations at pressures greater than one atmosphere, very large-sized equipment was required and nitric acid strength was limited to approximately 50 percent concentration. With the advent of acid resistant alloys capable of operating at higher pressures and the desirability to produce higher strength acids, industry decided to construct pressure or combination (dual-pressure) process plants.[1,2,3,8]

A description of each type of process for weak acid production is presented in the following sections.

3.1.3.1 Atmospheric Pressure Process--

It is very improbable that any new atmospheric process plants will be built. Although some of these early plants are still in existence, they are generally used only in a standby capacity. The atmospheric pressure process shown in Figure 7 is presented simply as a basis for understanding the other more common processes, because they can be considered derivatives of the atmospheric pressure process.

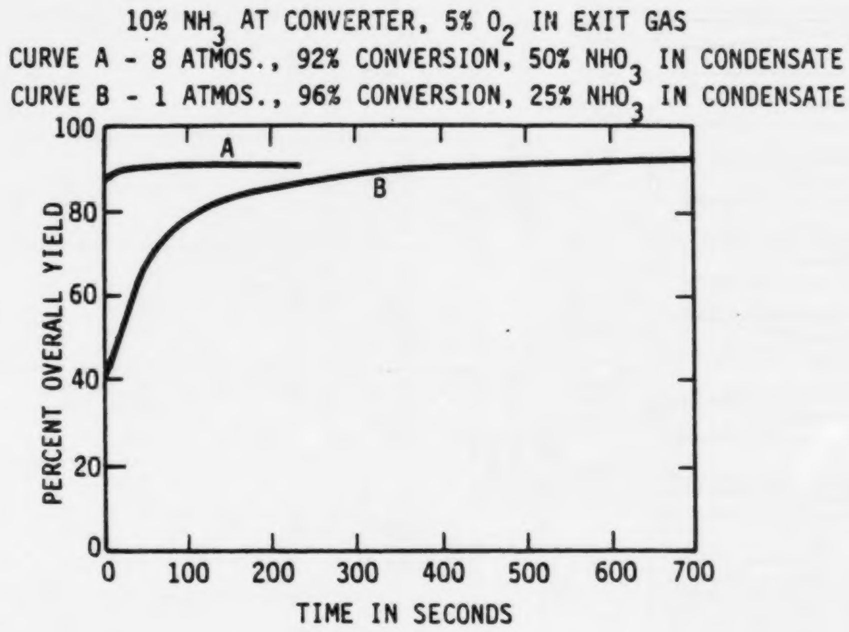
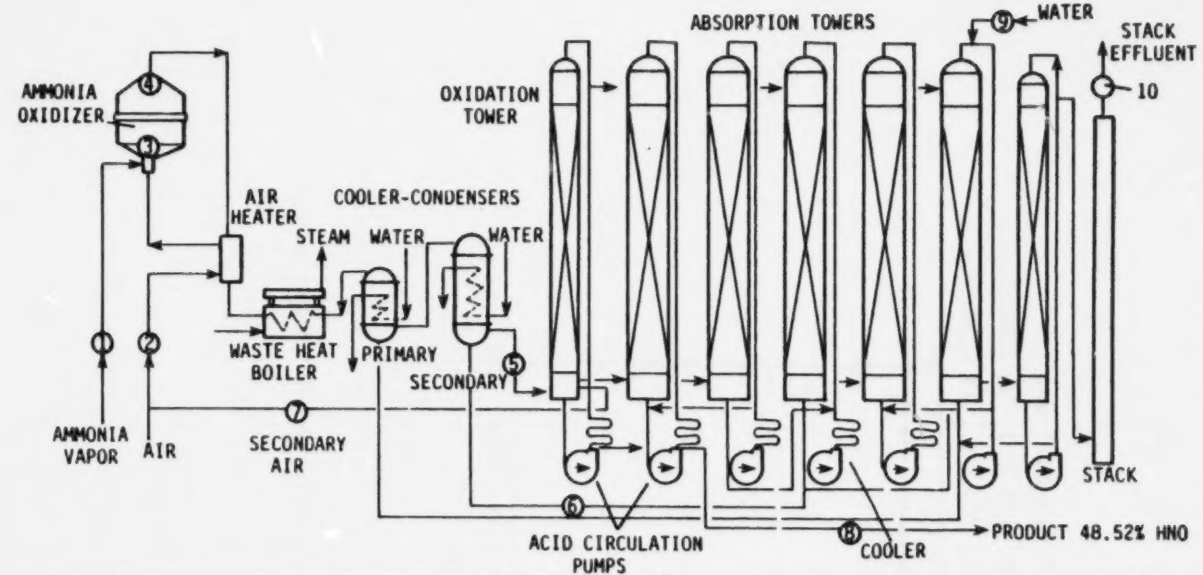


Figure 6. Minimum time for absorption in water of nitrogen oxides from ammonia oxidation.[1]


AMMONIA OXIDIZER
AIR HEATER
COOLER-CONDENSERS
STEAM
WATER
WATER
WASTE HEAT BOILER
PRIMARY
SECONDARY
OXIDATION TOWER
ABSORPTION TOWERS
WATER
STACK EFFLUENT
10
STACK
AMMONIA VAPOR
AIR
SECONDARY AIR
ACID CIRCULATION PUMPS
COOLER
PRODUCT 48.52% HNO
1
2
3
4
5
6
7
8
9

	1 AMMONIA	2 AIR	3 MIX	4 CONVERTER PRODUCTS	5 COOLED GAS	6 CONDENSATE ACID	7 SECONDARY AIR	8 NITRIC ACID PRODUCT	9 WATER	10 STACK EFFLUENT
FLOW, lb/h	2,850	54,250	46,100	46,100	44,350	1,750	11,000	18,850	6,750	45,000
TEMPERATURE, °F	100	60	270	1,550	95	95	60	80	100	85
PRESSURE, psig	5	5	5	4	3				40	0
NOH_3 Vol %	100		10.5	0	0					
NO Vol %				9.6	6.4					0.1
NO_2 Vol %				0	4.0	Nil				0.2
O_2 Vol %		20.8	18.7	6.2	4.7		20.8			4.0
H_2O Vol %		0.9	0.8	15.4	5.6		0.9			4.2
HNO_3 Wt %						25.7		50.0		
H_2O Wt %	<0.5					74.3		50.0	100	
H_2 Wt %		78.3	71.5	68.8	79.3		78.3			91.5

Figure 7. Flow diagram of a typical 120-ton-per-day nitric acid plant using the atmospheric pressure process.

In the atmospheric process, ammonia oxidation and conversion to nitric acid of the resultant nitrogen oxides is carried out at pressures near atmospheric. Briefly, this process includes the following steps.

Step 1. Liquid ammonia is evaporated and the resulting gas is collected and pumped by a fan to a mixer where filtered air is added. The resultant mixture of ammonia and air, usually containing from 9.5 to 11 percent ammonia, is brought in contact with the catalyst, where the ammonia is oxidized to form nitrous oxide and water.

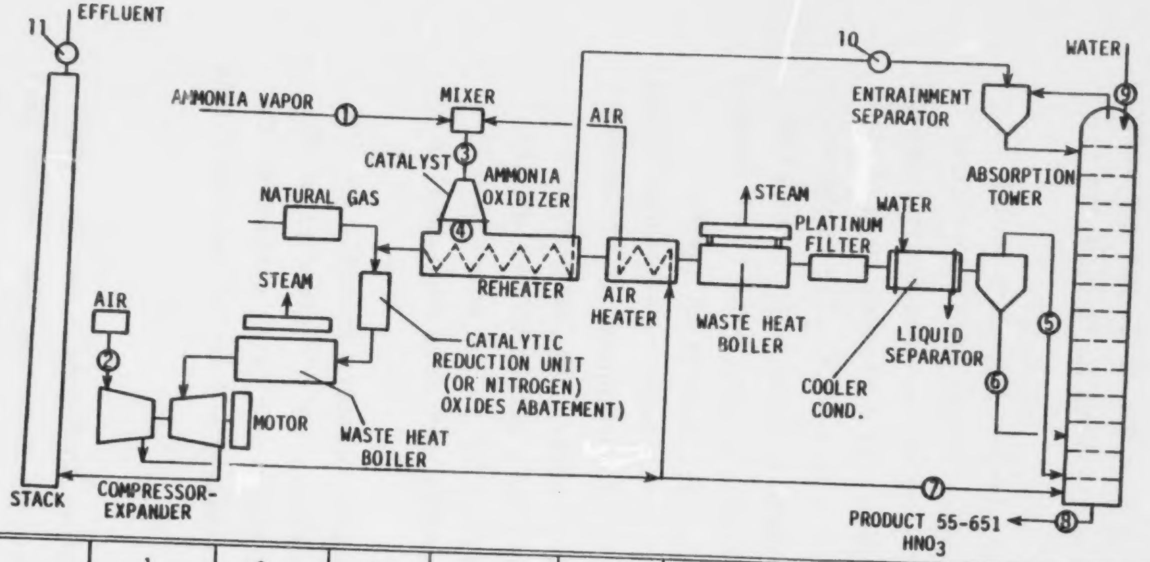
Step 2. After leaving the catalyst, the nitrous gases are cooled in the air preheater and the waste heat boiler where nitric acid is oxidized to NO_2. The gases are further cooled in a series of cooler-condensers in which nitric acid begins to form. The cooler-condenser will produce an acid containing approximately 20 to 22 percent nitric acid (HNO_3). The condensate formed during this step is conveyed to the absorption section of the plant where it is passed through a series of absorption towers. The final product (45 to 52 percent HNO_3) is drawn off from the last absorption tower.

Considering current technology, the main advantages of atmospheric-type plants are the lower platinum consumption and higher conversion efficiency from ammonia to nitric oxide in the burner. However, the improvement of platinum filter design, the efficiency of pressure burner units, and the greater absorption efficiency of pressure processes in the production of increased strength acid, make pressure processes much more economically attractive.

It should be noted that in the atmospheric pressure process, the low oxygen content of the tail gas and the low absorption efficiencies of NO_2 require a large number of towers to reduce the nitric oxide content of the tail gas to the acceptable figure of approximately 0.2 to 0.3 percent nitric oxide.

3.1.3.2 The Pressure Process--

The typical pressure process plants shown in Figure 8 are the result of technical advances in nitric acid production since the 1940 through 1950 time period. During World War II many of the pressure process plants were built to manufacture munitions, but these facilities did not have catalytic reduction units or turbine expanders. The first pressure-type plants operated


11
EFFLUENT
STACK
AMMONIA VAPOR
①
MIXER
③
CATALYST
AMMONIA OXIDIZER
④
NATURAL GAS
REHEATER
AIR
②
STEAM
CATALYTIC REDUCTION UNIT (OR NITROGEN) OXIDES ABATEMENT)
WASTE HEAT BOILER
MOTOR
COMPRESSOR-EXPANDER
AIR
AIR HEATER
STEAM
WASTE HEAT BOILER
PLATINUM FILTER
WATER
COOLER COND.
LIQUID SEPARATOR
⑥
⑤
⑦
10
ENTRAINMENT SEPARATOR
WATER
⑨
ABSORPTION TOWER
⑧
PRODUCT 55-651 HNO3

	1 AMMONIA	2 AIR	3 MIX	4 CONVERTER PRODUCTS	5 COOLED GAS	6 CONDENSATE ACID	7 SECONDARY AIR	8 NITRIC ACID PRODUCT	9 WATER	10 TAIL GAS	11 STACK EFFLUENT
FLOW, lb/h	2,900	51,600	47,050	47,050	38,350	8,700	7,450	16,650	3,350	41,200	42,600
TEMPERATURE, °F	170	60	450	1,650	105	105	450	135	100	85	450
PRESSURE, psig	170	0	112	112	90	98	120	95	125	92	0
NOH_3 Vol %	100	-	100	0	0	-	-	-	-	-	-
NO Vol %	-	-	-	9.3	1.1	-	-	-	-	0.10	0.01
NO_2 Vol %	-	-	-	0	6.4	1.2	-	-	-	0.15	Nil
H_2O Vol %	-	0.9	0.8	15.4	0.9	-	0.9	-	-	0.6	3.8
HNO_3 Wt %	-	-	-	-	-	40-50	-	60	-	-	-
H_2O Wt %	0.05	-	-	-	-	50-60	-	40	-	-	-
H_2 Wt %	-	78.3	17.5	69.0	91.6	-	78.3	-	-	96.18	94.2
CO_2 Vol %	-					-		-	-		2.0

Figure 8. Flow diagram of a typical 120-ton-per-day nitric acid plant using the pressure process.

at approximately 80 to 130 pounds per square inch absolute (psia) and had reciprocating compressors that recovered about 40 percent of the compressor power. In the 1950's the development of high temperature designs for gas turbines permitted improved power recovery and the use of centrifugal compressors permitted the production of air hot enough to be used directly in the converter. A catalytic reduction unit was added to the process to further improve power recovery. By heating the tail gas to approximately 1250°F before expansion, the catalytic reduction unit has resulted in the recovery of all the power required for air compression. The side effect of reducing the nitrogen oxide concentration in the tail gas was an added advantage as air pollution regulations have been broadened in recent years to include the control of nitrogen oxide emissions.

The pressure process, in which both ammonia oxidation and conversion of the resultant nitric oxides are carried out at greater than atmospheric pressure, consists of the following steps.

Step 1. Liquid ammonia is evaporated either directly from the heats of formation of nitrous gases or nitric acid, or indirectly by steam generated from the recovery of the heats of reaction in waste heat boilers.

Step 2. Air is compressed to a pressure that varies somewhat from plant to plant but in the most recent plants, is approximately 110 pounds per square inch gauge (psig). In large capacity units, the compressor used is generally of the centrifugal type.

Step 3. The streams of filtered ammonia gas and air mix and the mixture then goes to the burner. Air, delivered by centrifugal compressors, is usually hot (450° to 500°F) enough to avoid preheating. If reciprocal-type compressors are used, however, the air must be preheated. In both cases, the temperature of the air before being mixed with ammonia must be approximately 500°F. In the burner itself, the temperature at the catalyst is maintained at approximately 900°C (1,650°F).

Step 4. If power recovery is adopted, the gas leaving the burner passes through a heat exchanger to lower the tail gas temperature to approximately 950°F. The burner gas is then cooled at a pressure of 150 to 225 psig. A catalyst recovery filter is usually installed at the cold end of the boiler. Finally, the gas is cooled in a cooler-condenser in which most of the water produced by the oxidation reaction is removed as 40 to 45 percent nitric acid.

Step 5. The gas then flows into the absorption system where the oxidation of NO to NO_2 is then completed and these oxides are absorbed in water to form nitric acid.

Step 6. Weak acid from the condenser is introduced at an appropriate point in the absorption system. The absorption system proper may consist of one bubble-cap tray or of a cascade of absorption drums, each equipped with one tray and several coolers. In the bubble-cap tray tower, the coolers are placed directly on the trays around the bubble-caps.

Step 7. The product acid is bleached in the lower section of the absorption tower itself or in a separate bleacher. The acid produced (57 to 60% HNO_3) does not contain more than 0.05 percent nitrous acid.

The overall efficiency of absorption under pressure is sufficiently high (98 to 99%) to permit the elimination of alkali scrubbers.

The main advantage of the pressure type plant when compared with the atmospheric pressure type plant of the same efficiency is a significant reduction in equipment volume required for oxidation, heat exchange, and most importantly, absorption.

3.1.3.3 Combination Process--

In the combination process for nitric acid production, ammonia oxidation takes place near atmospheric pressure while absorption occurs at pressures much higher than atmospheric pressure, thereby using the optimum economic efficiencies for both conversion and absorption. The oxidation of ammonia typically occurs at pressures from atmospheric to 50 psia. The resulting nitrogen oxides are then cooled and compressed to approximately 50 to 215 psia for absorption. The lower conversion (oxidation) pressure gives the highest conversion efficiency at a lower operating temperature (1500^{o}F) which prolongs the life of the catalyst by a factor of 3 to 5 over the pressure process. The high pressure absorption, using smaller equipment, gives a high absorption efficiency and higher strength acid. This process, popular in Europe, is finding increased popularity in the United States. Increasing cost for electrical power, catalyst material, and ammonia, coupled with increasingly stringent nitrogen oxide (NO_x) emission regulations are causing this type of plant to be given increased consideration.

In this type of plant (Figure 9), the nitrous gases leaving the atmospheric oxidizer pass through condensers where they are cooled to a temperature close to that of the cooling water. A stainless steel turbo-compresser compresses gases to a pressure that may vary from 50 to 215 psig according to the type of plant.

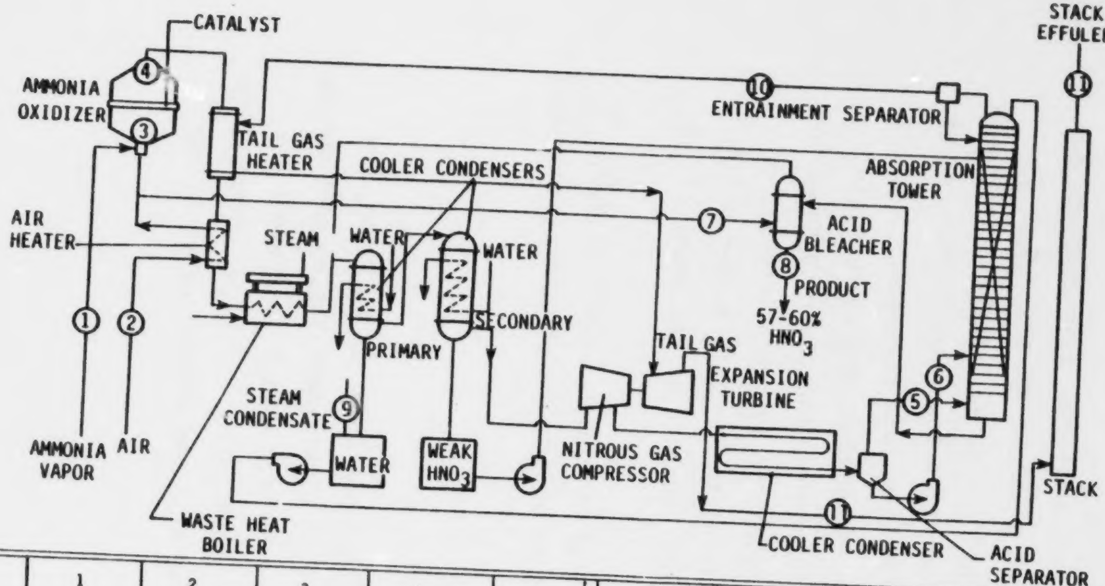
Because these nitrous gases contain an excess of air, this compression helps considerably in completing the oxidation of NO to NO_2 and N_2O_4. The compressor is followed by a condenser from which 33 to 42 percent nitric acid is withdrawn. This acid is pumped to the absorption tower and introduced at the level in the tower that corresponds to the same acid strength.

The acid condensed prior to compression is added at the appropriate points in the absorption system. In order to recover the power consumed for air compression, the tail gas leaving the absorption system is preheated by heat exchange with the hot nitrous gases leaving the ammonia burner before being released to the atmosphere by an expander mounted on the shaft of the gas compressor.

This combined scheme claims the advantage of higher ammonia oxidation yield and lower platinum loss, added to higher absorption efficiency and power recovery.

3.2 ATMOSPHERIC EMISSIONS

Ammonia oxidation nitric acid plants have one point of emission, the tail gas stack. The tail gas from nitric acid plants contains no particulate matter, and the temperature of the tail gas is high enough to prevent any mist formation as a result of water vapor condensation. Typical compositions of tail gas emissions from plants operating with and without controls are presented in Table 2.


CATALYST
AMMONIA OXIDIZER
④
③
TAIL GAS HEATER
⑩
ENTRAINMENT SEPARATOR
STACK EFFULENT
⑪
AIR HEATER
STEAM
COOLER CONDENSERS
WATER
WATER
PRIMARY
SECONDARY
⑦
ACID BLEACHER
⑧
PRODUCT
57-60% HNO3
ABSORPTION TOWER
①
②
AMMONIA VAPOR
AIR
STEAM CONDENSATE
⑨
WATER
WEAK HNO3
NITROUS GAS COMPRESSOR
TAIL GAS
EXPANSION TURBINE
⑤
⑥
⑪
WASTE HEAT BOILER
COOLER CONDENSER
ACID SEPARATOR
STACK

	1 AMMONIA	2 AIR	3 MIX	4 CONVERTER PRODUCTS	5 COOLED GAS	6 CONDENSATE ACID	7 SECONDARY AIR	8 NITRIC ACID PRODUCT	9 WATER	10 TAIL GAS	11 STACK EFFLUENT
FLOW, lb/h	2,850	53,550	45,680	45,680	52,730	3,700	10,750	16,650	3,600	43,450	43,450
TEMPERATURE, °F	170	60	270	1,550	160	95	250	80	60	85	450
PRESSURE, psig	5	5	5	4	40	40	5	0	50	40	0
NOH_3 Vol %	100		10.2	0	0						
NO Vol %				9.6	5.1					0.10	0.13
NO_2 Vol %				0	3.2	Nil				0.10	0.07
O_2 Vol %		20.8	18.7	6.2	8.0	20.8				4.0	4.0
H_2O Vol %		0.9	0.8	15.4	4.6	0.9				1.5	1.5
H_2 Wt %		78.3	70.3	68.8	79.1	78.3				94.3	94.3
HNO_3 Wt %						15.0		60.0			
H_2O Wt %	<0.5					85.0		40.0	100		

Figure 9. Flow diagram of a typical 120-ton-per-day nitric acid plant using the combination pressure process.

TABLE 2. TYPICAL COMPOSITIONS OF TAIL GAS EMISSIONS FOR NITRIC ACID PLANTS WITH AND WITHOUT CATALYTIC COMBUSTORS[9]

Tail gas emissions	Absorber exit	Catalytic combustor exit
Nitrogen oxide, ppm	1000-2000	40-350
Nitrogen dioxide, ppm	1000-2000	0-350
Oxygen, percent by volume	2-4	0-1.5
Temperature, $^{\circ}F$	90-100	1000-1300
Pressure, psig	85-95	75-90

The NSPS specify that NO_x emissions must not exceed: 1) 3.0 lb of nitrogen oxides expressed as NO_2 per ton of nitric acid (100 percent) produced (3-h average) and 2) a plume opacity of 10 percent. The emission limit of 3.0 lb of nitrogen oxides per ton of acid produced is equivalent to approximately 209 ppm (0.0209 percent by volume) concentration of nitrogen oxides in the process tail gas in a typical plant.

Nitric acid plants are primarily designed to achieve a specified production rate (tons of nitric acid in a water solution) using a given quality of ammonia feed. Other considerations necessary for the design of a plant are the specifications for certain auxiliary utilities such as cooling water, condensate quality water, boiler feed water, electricity, and process instrumentation.

When a plant is designed for a certain production rate, acid strength, and emission rate (lb NO_x per ton 100 percent acid), an increase in the production rate and/or acid strength will typically increase the NO_x emission rate. This is due to the fact that increasing the production rate decreases the time available for the oxidation of the nitrogen oxide to NO_2, decreases the absorption of the NO_2 resulting in efficiency losses and overloading of the catalytic reduction unit (if used), and increases the amount of NO_x emissions per ton of acid produced. On the other hand, if the plant is operated below the production rate and/or the acid strength is reduced, the time available for reactions and absorption is increased and a reduction of NO_x emissions would be expected.

3.2.1 Types of Emissions

3.2.1.1 Nitrogen Oxides--

Nitrogen oxide (NO_x) emissions consist mainly of NO and NO_2 and are emitted to the atmosphere in the absorber tail gas after it leaves the expander and final heat exchangers. These emissions vary with the plant design, maintenance practices, production rate, and absorber efficiency. Absorber efficiency can be increased by higher pressure and lower temperature. This will result in lower NO_x emissions. Emissions of NO_x from well-maintained and properly operated nitric acid plants without catalytic combustion devices or extended absorption average from 2000 to 3000 ppm. However, NO_x emissions as low as 1000 ppm have been reported at many existing plants. Catalytic combustion devices can reduce the NO_2 content significantly by reducing the NO to N_2. Catalytic combustion can also aid in energy recovery. Because NO_2 is the cause of the reddish-brown plume that may be observed, the effectiveness of the catalyst combustion process is evidenced by the colorless tail gas stack conditions.

3.2.1.2 Acid Mist--

Acid mist emissions do not occur from a properly operated plant. Acid mist is present in the gases leaving the absorber. These emissions, however, are largely removed by centrifugal collectors or mesh mist collectors before the gases are heated prior to entering the catalytic combustor or turboexpander. Trace quantities of acid mist present in the tail gas are vaporized as the gas is reheated.

3.2.1.3 Visible Emissions--

Visible emissions of a reddish-brown color occur from uncontrolled nitric acid plants due to the NO_2 in the exit gas. The opacity of stack gases varies with the NO_2 content and the stack diameter. The approximate visible threshold concentration of NO_2 is given by the suggested equation:[10]

$$Co = \frac{2400}{d}$$

where

d = stack diameter in inches

Co = visible concentration, ppm

Thus for a 12-inch diameter exit stack, a visible emission would occur at an NO_2 concentration of approximately 200 ppm. With larger diameter stacks, the visible plume would occur at lower NO_2 concentrations. The acid production system and storage tanks are the only significant sources of visible emissions at most plants. The amount of emissions from storage tanks is considerably less than the emissions from the stack.

3.2.1.4 Reducing Compounds--

Through the application of catalytic reduction systems, the NO_x abatement of nitric acid plant tail gas can result in the production and emission of hydrocarbons, carbon monoxide, and other reducing compounds. These compounds are produced as a result of the reducing atmosphere in the catalytic system. Although little data are available on the subject, it is expected that for a given system, reducing compounds are produced in inverse proportion to NO_x. The relevance of these secondary emissions is not well-documented. To date, no attempt has been made to control the release of reducing compounds to the atmosphere.

3.3 EMISSION CONTROL METHODS

After the promulgation of the 3.0 lb of NO_x per ton of acid NSPS in 1971, catalytic reduction was the primary NO_x abatement system installed on new nitric acid plants until 1975. With the developing natural gas shortage in 1975 and the advent of a proven alternate NO_x control technology (extended absorption), the entire nitric acid NO_x control situation was radically changed. From 1975 through mid-1978, 50 percent of new nitric acid plants employed the extended absorption process for NO_x control. Since 1979, all but one of the new or modified nitric acid plants were designed to use extended adsorption.[11] Although the extended absorption process can achieve the NSPS level of NO_x control, it is not able to reduce emissions to the same level obtainable with catalytic reduction. Other methods used for NO_x control on nitric acid plants include wet scrubbing, chilled absorption, and molecular sieve adsorption. The various NO_x control methods are summarized in Table 3. Table 4 provides typical tail gas analyses from uncontrolled plants, plants employing catalytic reduction, and those using the extended absorption process.

TABLE 3. NO_x CONTROL METHODS FOR NITRIC ACID PLANTS[1,2]

Process	Methods	Impacts
Catalytic reduction (nonselective)	Reduce NO_x and O_2 with CH_4, H_2 or CO to form N_2, H_2O, and CO_2. Pt or Pd catalyst used.	Fuel penalty usually required. Operable at any pressure. Energy recovery possible.
Catalytic reduction (selective)	Reduce NO_x with NH_3 using a Pt catalyst to form N_2 and H_2O.	Energy recovery not possible. Operable at any pressure.
Extended absorption	Uses an additional absorption unit.	Increases yield of HNO_3. Requires inlet pressure of 760 KPa.
Wet chemical scrubbing	Scrubbing tail gases with urea, NH_3, NaOH, sodium carbonate or potassium permanganate.	Operable at any pressure, but performs better at high pressure.
Chilled absorption	NO_x solubility increased in chilled H_2O.	Usually cannot meet NSPS alone when plant operated at capacity.
Molecular sieve adsorption	Uses synthetic zeolite adsorbent/catalyst bed. NO recycled by thermal regeneration.	High energy and capital requirements. NO_x emissions <50 ppm.

TABLE 4. TYPICAL NITRIC ACID PLANT TAIL GAS EMISSION COMPOSITIONS (PERCENT BY VOLUME)[a]

Component or tail gas	Plant control method		
	Uncontrolled[a]	Catalytic reduction	Extended absorption[b]
NO	0.10	0.01	
NO_2	0.15	trace	0.015
N_2	96.15	94.20	96.00
O_2	3.00	trace	3.50
H_2O	0.60	3.80	0.50
CO_2		2.00	

[a] Conventional design has been 98 percent absorption efficiency (this composition is typical of the tail gas before catalytic reduction).

[b] This plant is controlled by increasing the absorption efficiency to 99.8 percent plus.

3.3.1 Catalytic Reduction[1,2]

3.3.1.1 Nonselective--

As illustrated in Figure 10, the nonselective catalytic reduction unit is an integral part of the nitric acid plant design when it is used. Converter effluent gas is used for preheating the tail gas from the absorption tower. In the catalytic unit, fuel is burned to generate heat and reduce NO_x emissions in the tail gas. Hot exhaust gases from the catalytic unit are directed to an expander to drive the ammonia converter process air compressor. To keep the expander inlet temperature below its design maximum, typically 677°C (1250°F), a waste heat boiler is sometimes used directly after the catalytic unit. Prior to venting the tail gas to the atmosphere, a waste heat boiler recovers energy from the expander outlet gas in the form of steam.

In nonselective reduction, absorber tail gases are mixed with a fuel such as methane, carbon monoxide, or hydrogen and heated to the required ignition temperature. Ignition temperatures range from 150°C to 200°C for carbon monoxide and hydrogen and from 480°C to 510°C for natural gas (methane). The absorber tail gas fuel mixture is passed to the catalytic reduction unit

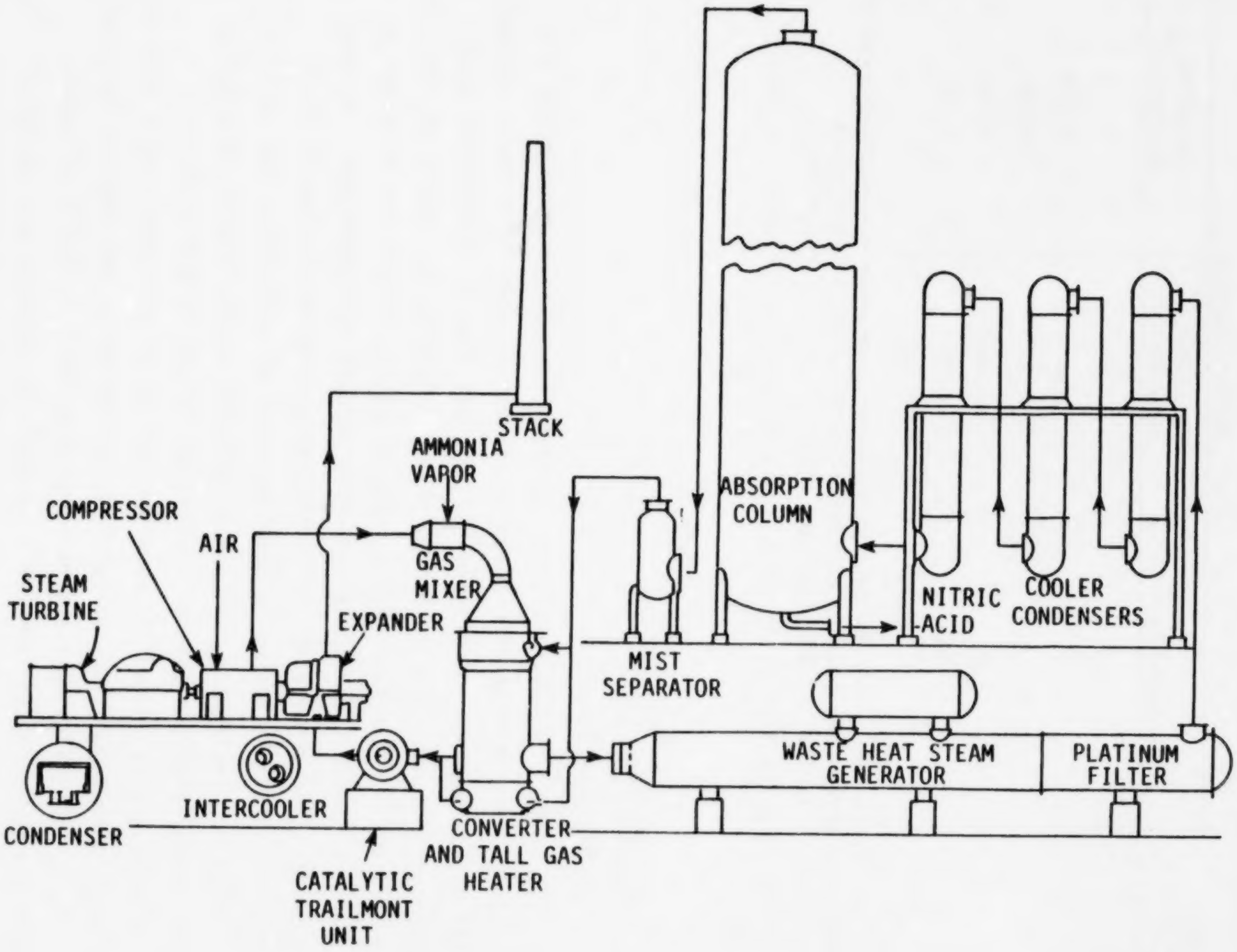

STACK
AMMONIA VAPOR
COMPRESSOR
AIR
STEAM TURBINE
GAS MIXER
EXPANDER
ABSORPTION COLUMN
NITRIC ACID
COOLER CONDENSERS
MIST SEPARATOR
WASTE HEAT STEAM GENERATOR
PLATINUM FILTER
CONDENSER
INTERCOOLER
CONVERTER AND TALL GAS HEATER
CATALYTIC TRAILMONT UNIT

Figure 10. Acid plant incorporating catalytic reduction for NO_x abatement.[9]

where the fuel reacts with NO_x and oxygen to form CO_2, water, and nitrogen over a 0.5 percent platinum or palladium catalyst. When methane (natural gas) is used for fuel, the following reactions occur:

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$$

$$CH_4 + 4NO_2 \rightarrow 4NO + CO_2 + 2H_2O$$

$$CH_4 + 4NO \rightarrow 2N_2 + CO_2 + 2H_2O$$

These reduction reactions are highly exothermic. The temperature rise resulting from catalytic reduction is 128°C for each percent oxygen burnout when methane is used as the fuel, and 150°C when hydrogen is employed. Due to the catalyst thermal limitation, the final reduction reaction must be limited to a temperature of 843°C (1550°F). It is interesting to note that the second reaction given above does not decrease total NO_x emissions; it simply converts NO_2 to colorless NO.

Although the catalyst thermal limitation is 843°C, the maximum temperature limit for the turbo-expanders is approximately 650°C (1200°F). Thus, absorber tail gas with greater than 2.8 percent oxygen will require cooling to prevent catalyst deactivation. Even greater cooling is required if power recovery is practiced. In the single-stage units, ceramic spheres are used as catalyst supports, at hourly gas space velocities up to 30,000 volumes per hour per volume. Single-stage units can only be used when the oxygen content of the absorber tail gas is less than 2.8 percent. The effluent gas from these units must be cooled by heat exchange or quenched to meet the temperature limitation of the turbine. Two-stage reduction units often employ honeycomb ceramic catalysts, with hourly space velocities of approximately 100,000 volumes per hour per volume in each stage.[3] One system uses interstage heat removal[4] while another feeds only 70 percent of the fuel to the first stage and uses the remaining 30 percent (preheated to only 250°F) for contact interstage cooling.[9,12,13] This second system eliminates the need for coolers or waste heat boilers. Historically, experience with single-stage reduction units has been more satisfactory than that obtained with two-stage units.

Usually, 90 to 95 percent of the nitrogen oxides in the tail gas are decomposed by nonselective catalytic reduction. Table 5 summarizes the typical operating conditions for both selective and nonselective reduction units. Fuel requirements for nonselective reduction with natural gas are typically 10 to 20 percent over stoichiometric, and some hydrocarbons and CO are discharged in the treated tail gas. Less surplus fuel is required when hydrogen is used. The particular process chosen is governed by a balance between capital costs and the availability and cost of fuels. Significant economic return, however, can be obtained by recovery of heat generated in the reduction unit, depending on the overall plant heat balance.

3.3.1.2 Selective--

In the selective catalytic reduction process, ammonia is added to absorber tail gas and the mixture is usually directed through a honeycomb support platinum or another type catalyst. The following reactions occur and reduce the nitrogen oxides to nitrogen:

$$8NH_3 + 6NO_2 \rightarrow 7N_2 + 12H_2O$$

$$4NH_3 + 6NO \rightarrow 5N_2 + 6H_2O$$

Proper operation of this process requires that the tail gas stream be maintained between 210°C and 270°C. Above 270°C, ammonia may be oxidized to NO_x and below 210°C, ammonium nitrate may be formed. This process can operate at any pressure. Many systems, however, operate at ambient pressure. User experience with this process has been good with catalyst lifetimes of over two years reported. The cost of ammonia, however, may not be competitive with other fuels even when less is required. On the other hand, the process does not require any gas stream cooling, and its lack of pressure sensitivity makes it applicable as a retrofit control device for existing low pressure nitric acid plants.

3.3.2 Extended Absorption[1]

Prior to promulgation of NSPS in 1971, most nitric acid plants were designed with absorption efficiencies of 98 percent and an emission rate of 15 to 17.5 kg of NO_x (as NO_2) per metric ton of nitric acid produced. Since 1975, increasing the design absorption efficiency has become the preferred

TABLE 5. OPERATING CONDITIONS FOR TREATING NITRIC ACID PLANT TAIL GAS BY CATALYTIC REDUCTION

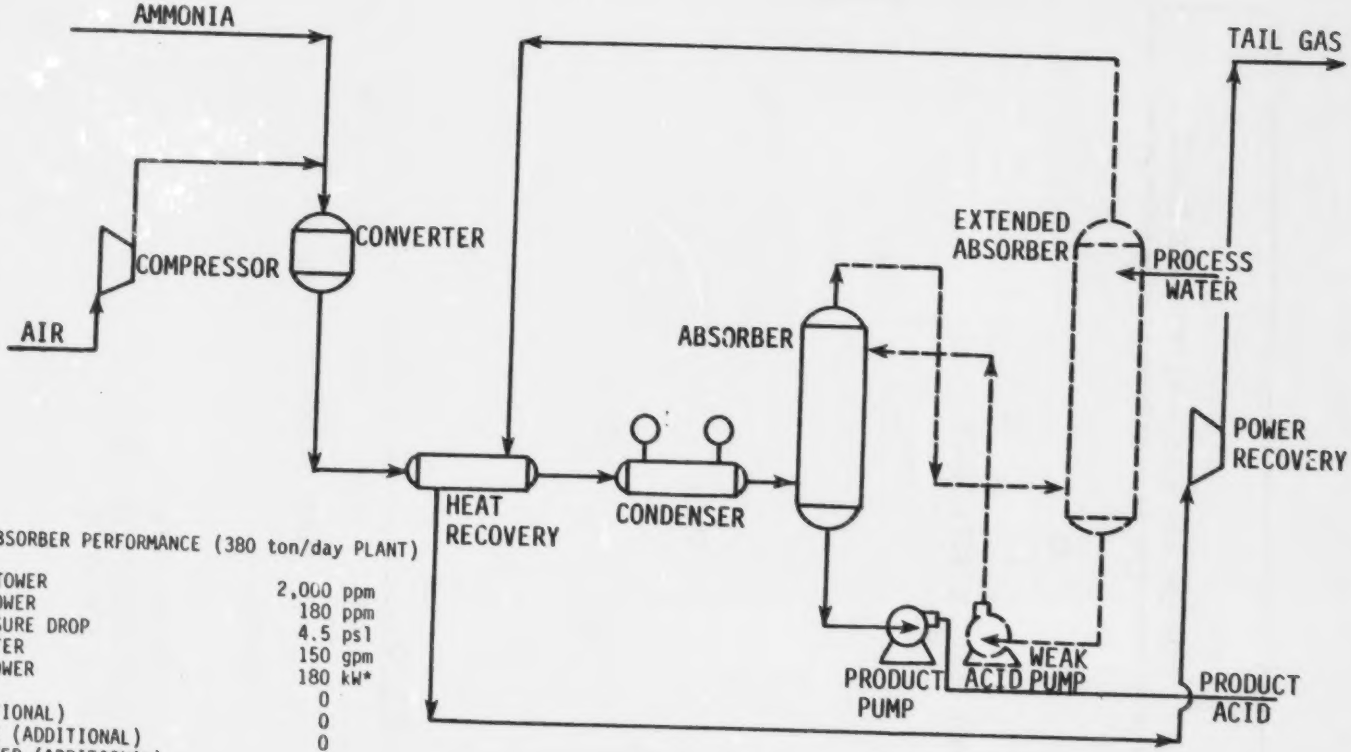
Process conditions	Nonselective catalytic reduction			Selective catalytic reduction
Usable fuels	Natural gas, hydrogen, coke oven gas, propane			Ammonia
Assumed fuel	Natural gas			Ammonia
O_2 in tail gas, %	2-3	3-5		2-5
Number of stages	1	2		
		1st stage	2nd stage	
Preferred catalyst	Pd	Pd	Pd	Pt
Space vel., SCFH/CF	60,000	20,000 to 40,000	60,000	30,000
Minimum inlet temperature, °C	460	440 to 500	460	180
Fuel, volume % of tail gas	1.1-1.7	0.8-1.3	0.9-1.4	0.3
Approximate temperature rise, °C	130 to remove all O_2 from tail gas containing 1% O_2			20-40

Source: Gillespie et al., 1972.

control method for NO_x emissions from new nitric acid plants. Thus the extended absorption process, in which a second absorption tower is added in series to the existing or first absorber, is not really an add-on control device for new plants but an integral component of the design for new nitric acid plants.

The Grande Paroisse version of the extended absorption process is illustrated in Figure 11 along with typical operating conditions. The nitrogen oxides in the tail gas from the first absorber is contacted with water in the second absorption tower to form additional nitric acid. The weak acid from the second absorber is recycled to the first. Thus, no liquid effluent is created by this process. Process economics usually require inlet gas pressures at the absorber of at least 730 Kilopascals (kPa) or 107 psig, and cooling of the tail gas is generally required if the inlet NO_x concentration is greater than 3000 ppm.[8] The D.M. Weatherly Company's version of the extended absorption process uses cooling water refrigeration to decrease the required volume and number of trays in the second absorption tower. In this version of the process, two cooling water systems are used for cooling the absorbers. The entire second absorber and approximately one-third of the trays of the first tower are cooled with water at approximately $7^{o}C$ ($45^{o}F$). Other trays in the first absorber are cooled with normal plant site cooling water, which is generated by mechanical refrigeration. This refrigeration process is a part of the ammonia vaporization section of the nitric acid plant.

The extended absorption process cannot control NO_x emissions to the same degree as that obtained with catalytic reduction, and this process frequently allows NO_x emissions greater than NSPS levels during startup and shutdown and during periods of high ambient temperatures unless the plant is specifically designed to compensate for these periods. Figure 12 illustrates the results of NSPS performance tests conducted on new nitric acid plants from 1971 to 1978. Table 6 summarizes the results of compliance tests obtained from 10 new nitric acid plants that have been constructed since 1979. A review of Figure 12 and Table 6 indicates that plants controlled by the extended absorption process generally had higher NO_x emissions. All design conditions must be precisely controlled for the extended absorption process to operate within NSPS emission limits. Absorber pressure, temperature, and the oxygen and NO_x content of the tail gas must remain within design limits. Table 7 shows


AMMONIA
AIR
COMPRESSOR
CONVERTER
HEAT RECOVERY
CONDENSER
ABSORBER
EXTENDED ABSORBER
PROCESS WATER
TAIL GAS
POWER RECOVERY
PRODUCT PUMP
WEAK ACID PUMP
PRODUCT ACID

EXTENDED ABSORBER PERFORMANCE (380 ton/day PLANT)

NO_x INLET TOWER	2,000 ppm
NO_x EXIT TOWER	180 ppm
TOWER PRESSURE DROP	4.5 psi
COOLING WATER	150 gpm
ELECTRIC POWER	180 kW*
CATALYST	0
FUEL (ADDITIONAL)	0
MAINTENANCE (ADDITIONAL)	0
PROCESS WATER (ADDITIONAL)	0
OPERATIONS MAN HOURS (ADDITIONAL)	0
ADDITIONAL PRODUCTION	8.1 tons/day

*88 kW REQUIRED TO OFFSET 4.5 psi PRESSURE DROP.

Figure 11. Extended absorption system for NO_x emissions control.[11]

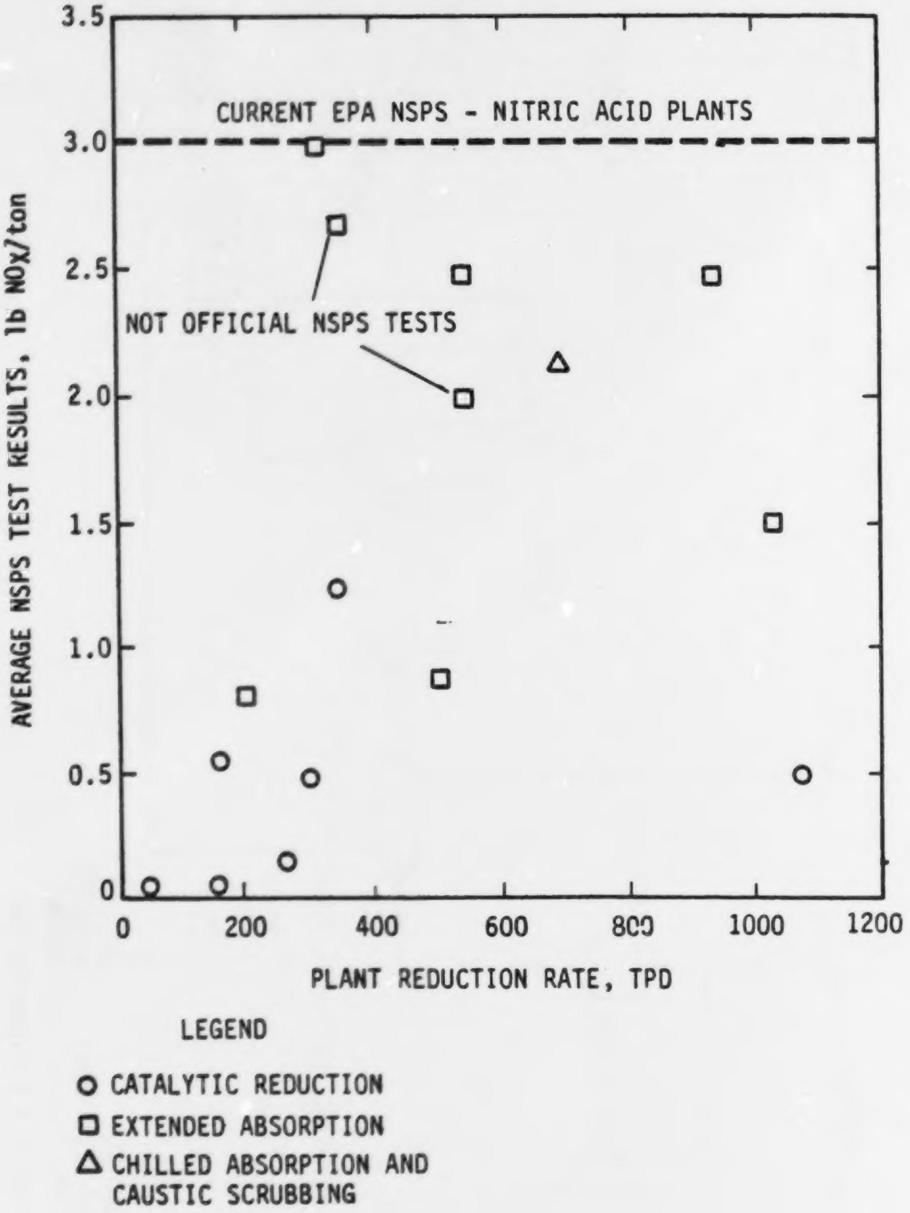


Figure 12. NSPS test results of NO_x emissions at nitric acid plants.[15]

TABLE 6. COMPLIANCE TEST RESULTS FOR NITRIC ACID PLANTS SUBJECT TO THE NSPS SINCE THE 1979 REVIEW[11]

Plant	Control technique	Average NO_x emissions, lb/ton
A	Chilled absorption & caustic scrubbing	1.84
B	Catalytic reduction	1.13
C	Extended absorption	1.3
D	Extended absorption	2.75
E	Extended absorption	1.8
F[a]	Extended absorption	4.1
G	Extended absorption	2.55
H	Extended absorption	2.81
I	Extended absorption	2.74
J	Extended absorption	2.13
	NSPS =	3.0

[a] Plant was tested upon startup, then shut down, and has not restarted.

TABLE 7. EXCESS EMISSIONS DATA FROM EXTENDED ABSORPTION NITRIC ACID PLANT[14] EXPERIENCING OPERATING PROBLEMS[a,b]

Date	Time period	Emissions, lb NO_x per ton of 100% HNO_3	Cause
7/2/78	12-1 pm 1-2 pm 2-3 pm 3-4 pm	3.83 4.52 4.26 3.52	Startup
7/11/78	6-7 pm 7-8 pm 8-9 pm	5.89 4.99 4.15	Startup followed shutdown due to expander bypass valve malfunction
7/23/78	2-3 pm 3-4 pm 4-5 pm	5.71 6.65 3.76	Startup
8/7/78	3-4 pm 4-5 pm 5-6 pm 6-7 pm 7-8 pm	4.27 4.23 3.27 5.20 3.48	Startup followed shutdown due to high gauge temperature trip
8/16/78	6-7 pm 7-8 pm 8-9 pm	4.25 5.65 2.32	Startup followed shutdown due to NO_x compressor trip
8/20/78	11-12 am 12-1 pm 1-2 pm 2-3 pm	2.81 3.29 3.40 3.07	Reduced absorption tower efficiency due to high ambient temperature
8/22/78	11-12 am 12-1 pm 1-2 pm 2-3 pm	2.88 3.23 3.24 3.00	Reduced absorption tower efficiency due to high ambient temperature
8/24/78	1-2 pm 2-3 pm 3-4 pm	5.49 4.17 3.74	Startup followed shutdown due to NO_x compressor trip
8/25/78	1-2 pm 2-3 pm 3-4 pm	4.25 2.89 2.46	Startup followed shutdown due to mysterious trip
8/29/78	5-6 pm 6-7 pm 7-8 pm	3.23 2.63 3.55	Startup followed shutdown due to electrical power failure

[a] Increased emissions occur during startups due to lag time in establishing required absorption tower pressure and lowered circulating water temperature. No external NO_x abatement system is used at this acid plant.

[b] Pounds of NO_x are calculated based on 225 ppm(v) equal to 3 lb/ton at 100 percent production rate.

the NO_x emissions from one plant controlled by the extended absorption process during unstable operating conditions and times of high ambient temperatures. Unstable operating conditions must result in high NO_x emissions with this process because no external control device is available in these situations to reduce emissions. Ambient temperatures of 95^0 to 100^0F create higher NO_x vapor pressures than the second absorber can handle. For this reason, one vendor will only guarantee performance within the specified NO_x emission limit 95 percent of the time for plants located in the southern tier of States.[1]

Recent trends indicate that the extended absorption process will remain the preferred NO_x control technology at new nitric acid plants for the next several years. Of the nine extended-absorption process-controlled new nitric acid plants that came on stream through mid-1978, eight came on stream since the energy crisis of the mid-1970's. This control technology choice appears to have been influenced by the increasing uncertainty of an adequate natural gas supply (the principal fuel used for catalytic reduction) together with the anticipated sharp increases in natural gas prices over the next several years. The economics of catalytic reduction, however, can be more favorable than those of extended absorption if the nitric acid plant is to be located in a fertilizer complex with an available contract for a low-price, long-term natural gas supply.[1]

3.3.3 Wet Chemical Scrubbing

Wet scrubbing consists of contacting absorber tail gas with alkali hydroxides or carbonates, ammonia, urea, or potassium permanganate solutions to absorb NO_x in the form of nitrate and/or nitrite salts. Although these processes have traditionally been used for retrofit control of existing nitric acid plants, at least one nitric acid plant subject to NSPS employs a combination of chilled absorption and caustic scrubbing for NO_x control.[1] Some of these processes create potentially serious water pollution problems, and if a market cannot be found for recovered ammonium nitrate, there is also a solid waste disposal problem. Nitrogen oxide control, through use of wet chemical scrubbing, has generally proved to be adequate.

3.3.3.1 Urea Scrubbing[16]--

The MASAR process serves as a representative example of urea scrubbing and is illustrated in Figure 13. The process control device consists of a three-stage absorption column with gas and liquid chillers on the feed gas and recirculated solvents. Liquid ammonia or some other form of mechanical refrigeration is used as the cooling medium. The chemical reaction mechanisms proposed for urea scrubbing are as follows:

$$HNO_2 + CO(NH_2)_2 \rightleftarrows N_2 + HNCO + 2\ H_2O$$

$$HNCO + HNO_2 \rightleftarrows N_2 + CO_2 + H_2O$$

$$HNCO + H_2O + H+ \rightleftarrows NH_4 + CO_2 \text{[17]}$$

Under actual process operating conditions, the last reaction listed above predominates so that the overall reaction is:

$$HNO_2 + CO(NH_2)_2 + HNO_3 \rightarrow N_2 + CO_2 + NH_4NO_3 + H_2O$$

This process has been reported to reduce NO_x emissions from 4000 to 100 ppm and can theoretically be designed for no liquid effluent. In practice, however, liquid blowdown of 16 kg/h (35 lb/h) of urea nitrate in 180 kg/h (396 lb/h) of water is estimated for a plant with a capacity of 320 Mg of acid/day (350 tons/day).

In the MASAR process, absorber tail gas is first cooled in a gas chiller where condensation occurs with the formation of nitric acid. Normal plant absorber feedwater is chilled in Section C of the MASAR absorber and is then fed to Section A, where it flows countercurrent to the incoming chilled tail gas in the packed bed. After additional NO_x is scrubbed from the tail gas, the scrubbing water is recirculated through a chiller to remove reaction heat; this weak acid stream is used as feed to the nitric acid plant absorber. In Section B of the MASAR absorber, the tail gas is scrubbed with the urea-containing solution forming nitric acid and nitrous acid that reacts to form $CO(NH_2)$, N_2 and H_2O. Recirculation of the scrubbing solution causes the concentration of nitric acid and ammonium nitrate to rise. Therefore, a bleed

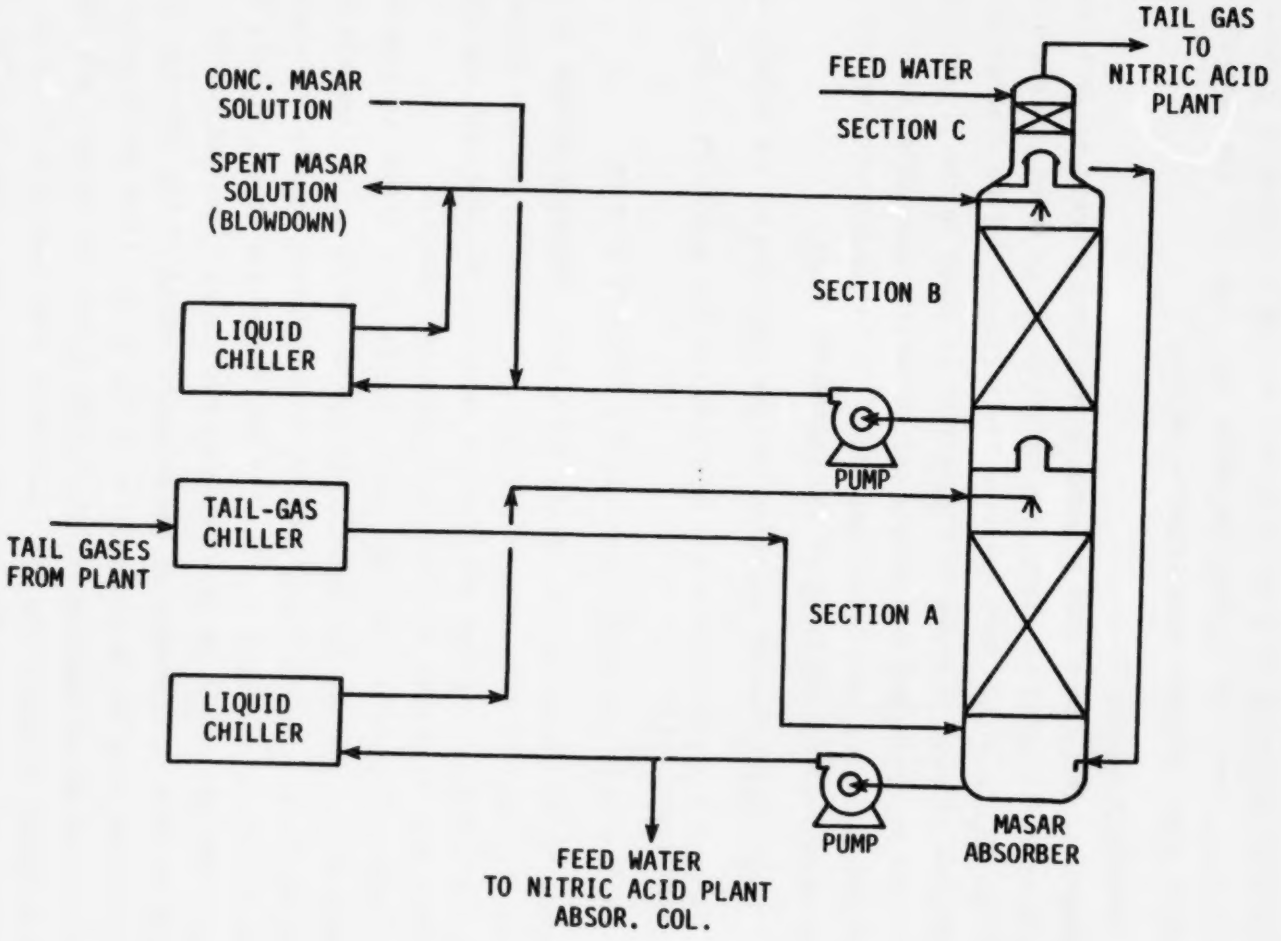

TAIL GAS TO NITRIC ACID PLANT
FEED WATER
SECTION C
CONC. MASAR SOLUTION
SPENT MASAR SOLUTION (BLOWDOWN)
SECTION B
LIQUID CHILLER
PUMP
TAIL-GAS CHILLER
TAIL GASES FROM PLANT
SECTION A
LIQUID CHILLER
PUMP
MASAR ABSORBER
FEED WATER TO NITRIC ACID PLANT ABSOR. COL.

Figure 13. Flow diagram of the MASAR process.[16]

stream is required to keep the system in balance. Makeup urea/water solution is fed to the scrubbing system at a rate sufficient to maintain a specified minimum urea residual content. To maintain temperature control in Section B, the recirculated scrubbing solution is pumped through a chiller to remove the heat of reaction. Prior to leaving the MASAR unit, the tail gas is again scrubbed with plant absorber feed water in Section C.

3.3.3.2 Ammonia Scrubbing[18]--

Goodpasture, Inc., developed an ammonia scrubbing process in 1973 that is suitable to retrofit existing plants for reduction of an inlet concentration of 10,000 ppm NO_x to within NSPS emission limitations. A flow diagram for the Goodpasture process is shown in Figure 14. As shown in the figure, the entire process is conducted in a single packed contact absorption tower with three sections operated in a concurrent flow. In the Goodpasture process, there are three distinct sections of the absorption tower:

1. A gas absorption and reaction section operating on the acidic side.
2. A second gas absorption and reaction section operating on the ammoniacal side.
3. A final mist collection and ammonia recovery section.

Feed streams to this process are ammonia and water. Ammonium nitrate is produced as a byproduct of this process. Successful operation of this process requires that ammonium nitrite formation be kept to a minimum, and any ammonium nitrite that does form must be oxidized to ammonium nitrate.

As shown in Figure 14, tail gas enters the first or acidic section of the tower where NO_x in the gas stream is converted to nitric acid. Ammonia is added to the process in the second section in sufficient amounts to maintain the pH at a level of 8.0 to 8.3. In this section of the tower, ammonia reacts with NO_x in the gas stream to form ammonium nitrate and ammonium nitrite; the proportion of each is dependent on the oxidation state of the nitrogen oxides. Product solution from the second section is fed to the first where ammonium nitrite is oxidized to ammonium nitrate by the acidic conditions, and ammonium nitrate is formed directly from the reaction of free ammonia with nitric acid. The resulting solution is split into two streams. One stream is withdrawn from the process as product solution, while the other is fed to the second or ammoniacal section of the tower. Feed streams to the third and final section

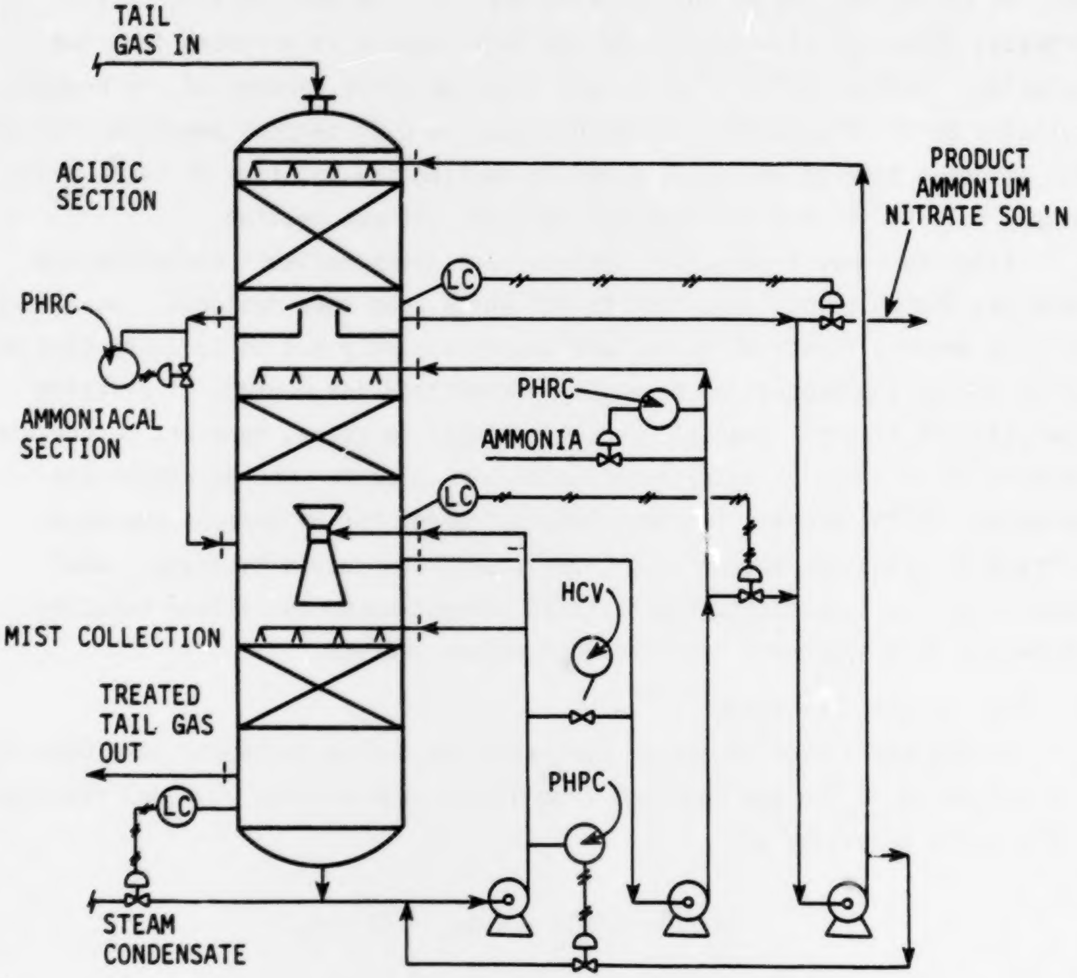

TAIL GAS IN
ACIDIC SECTION
PRODUCT AMMONIUM NITRATE SOL'N
LC
PHRC
PHRC
AMMONIA
AMMONIACAL SECTION
LC
HCV
MIST COLLECTION
TREATED TAIL GAS OUT
LC
PHPC
STEAM CONDENSATE

Figure 14. Process flow diagram for the Goodpasture process.[18]

of the tower consist of process water or steam condensate in sufficient quantities to maintain the product ammonium nitrate solution in the 30 to 50 percent concentration range, and a small amount of solution from the acidic section to control the pH to approximately 7.0. In this section of the process, mists are eliminated, and any free ammonia is stripped from the solution. Product solution withdrawn from the first section of the process contains 35 to 40 percent ammonium nitrate and 0.05 percent ammonium nitrite. The ammonium nitrite can be oxidized by heating the solution to $240^{\circ}F$ or by simply holding it in a day-tank for 24 hours without heating.

Existing ammonia scrubbing systems have given reliable operation and have met the emissions requirements for which they were designed. An advantage of this process is that the pressure losses are only 6.8 to 13.0 kPa (1-2 psi), which allows the process to be easily retrofitted for control of existing low-pressure plants. Special precautions must be taken, however, to prevent deposition of ammonium nitrate on the turbine blades. One potential disadvantage of the process is that the requirement for 85 percent ammonium nitrate solutions by modern fertilizer plants can necessitate additional evaporators to concentrate the 35 to 55 percent ammonium nitrate solution recovered as a byproduct from the Goodpasture process.

3.3.3.3 Caustic Scrubbing[15,19]--

Strong bases such as sodium hydroxide and sodium carbonate have been used for scrubbing NO_x in the tail gas from nitric acid plants. Typical reactions for caustic scrubbing are:

$$2NaOH + 3NO_2 \rightarrow NaNO_3 + NO + H_2O$$

$$2NaOH + NO + NO_2 \rightarrow 2NaNO_2 + H_2O$$

One serious problem with this process is that disposal of the spent scrubbing solution can create a serious water pollution problem. At least one nitric acid plant subject to NSPS, however, has successfully employed a combination of caustic scrubbing and chilled absorption for NO_x control.

3.3.3.4 Potassium Permanganate Scrubbing[19]--

Several Japanese plants employ solutions of potassium permanganate for scrubbing NO_x emissions, but no U.S. plants are known to use the process. In this process, permanganate is reduced to manganate that must be electrolytically oxidized. The cost of electrolysis along with permanganate makeup costs are believed to make this process uneconomical in the United States.

3.3.4 Chilled Absorption[20]

The chilled absorption process exploits the increased solubility of NO_x in cold water. This method has been primarily used as a retrofit for existing plants, and as a sole control device, it is not capable of meeting NSPS emission limits. Both water and brine have been used in a closed loop system to provide local cooling to the liquid on the trays of the absorption tower. One representative chilled absorption process is the CDL/VITOK process, which is illustrated in Figure 15. Nitrogen oxide in the tail gas is both chemically oxidized and physically absorbed as it is contacted with a nitric acid solution upon entering the absorber. In the upper portion of the absorber, cooled water is employed to improve absorption. Nitric acid solution from the absorber is split off to a bleacher where further oxidation occurs and air removes entrained gases. Bleached nitric acid solution can either be stored or recycled to the absorber after the addition of makeup water. A closed loop system is used to chill both recirculated acid and cooling water by ammonia evaporation. Variations in the CDL/VITOK process include addition of an auxiliary bleacher operating in parallel with the primary unit and use of a secondary absorber with its own bleacher.

3.3.5 Molecular Sieve Adsorption[21,22]

Although the molecular sieve process has been somewhat successful in controlling NO_x emissions from existing plants, there have been no new nitric acid plants built that are subject to NSPS which employ this form of NO_x control.[1] Principal objections to the process are high capital and energy costs, the problems of coupling a cyclic system to a continuous acid plant operation, and bed fouling. In addition, molecular sieves are not considered as state-of-the-art technology for NO_x control in nitric acid plants.[15,19] A flow diagram of the Purasiv control system that was developed by Union

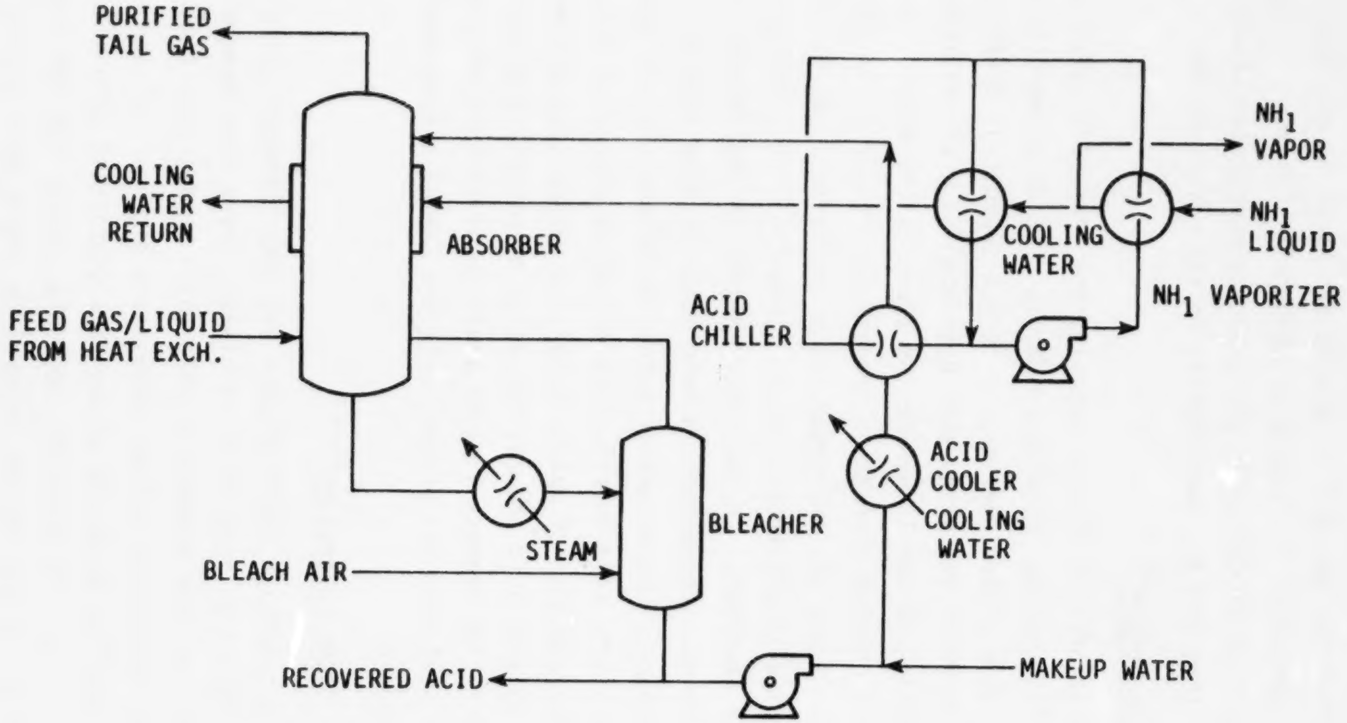

PURIFIED TAIL GAS
COOLING WATER RETURN
FEED GAS/LIQUID FROM HEAT EXCH.
ABSORBER
STEAM
BLEACH AIR
BLEACHER
RECOVERED ACID
ACID CHILLER
ACID COOLER
COOLING WATER
MAKEUP WATER
COOLING WATER
NH1 VAPOR
NH1 LIQUID
NH1 VAPORIZER

Figure 15. Schematic flow sheet of the CDL/VITOK NO_x removal process.[20]

Carbide is shown in Figure 16. The pressure drop through the sieve bed is rather high and averages 34 kPa (5 psi). The average concentration of NO_x in the treated tail gas discharged to the atmosphere, however, is only 50 ppm.

The fundamental principle behind molecular sieve control is selective adsorption of NO_x followed by recycle of the NO_x back to the nitric acid plant absorption tower. As illustrated in Figure 16, the first step of the process is to chill the absorber tail gas to between 7^o and 10^oC; the exact temperature required is governed by the NO_x concentration in the tail gas stream. Next, the chilled gas is passed through a mist eliminator to remove entrained water and acid mist. Weak acid collected in the mist eliminator is either recycled to the absorption tower or stored. Partially dried tail gas then passes to the sieve bed where several operations proceed simultaneously:

1. Dessicant contained in the bed removes the remaining moisture from the gas stream.
2. NO in the tail gas is converted catalytically to NO_2.
3. NO_2 is selectively adsorbed.

Regeneration is accomplished by thermal-swinging (cycling) the adsorbent/catalyst bed after it is nearly saturated with NO_2. Regeneration gas is obtained by heating a portion of the treated tail gas in an oil- or gas-fired heater. This gas is then used to desorb NO_2 from the bed for recycle back to the nitric acid plant absorption tower. Both adsorption and regeneration of the bed require approximately four hours.

3.3.6 Emerging Technology[23]

The new Bolme nitrogen oxides recovery process recovers NO_x removed from tail gas and recycles it as feed to the plant absorption tower. When installed in a new medium-pressure plant, as part of an integrated nitric acid-NO_x recovery system, it can reduce total capital cost and yield a modest increase in operating profit. The process is illustrated in Figure 17 and design specifications are listed in Table 8. In essence, the process employs a 25 to 30 weight percent nitric acid solution for scrubbing NO_x in absorber tail gas, and then the recovered NO_x is steam stripped from the scrubbing solution and recycled. This process can be economically designed for NO_x emissions of only 70 to 85 ppm when operated at capacity during the hottest week of the

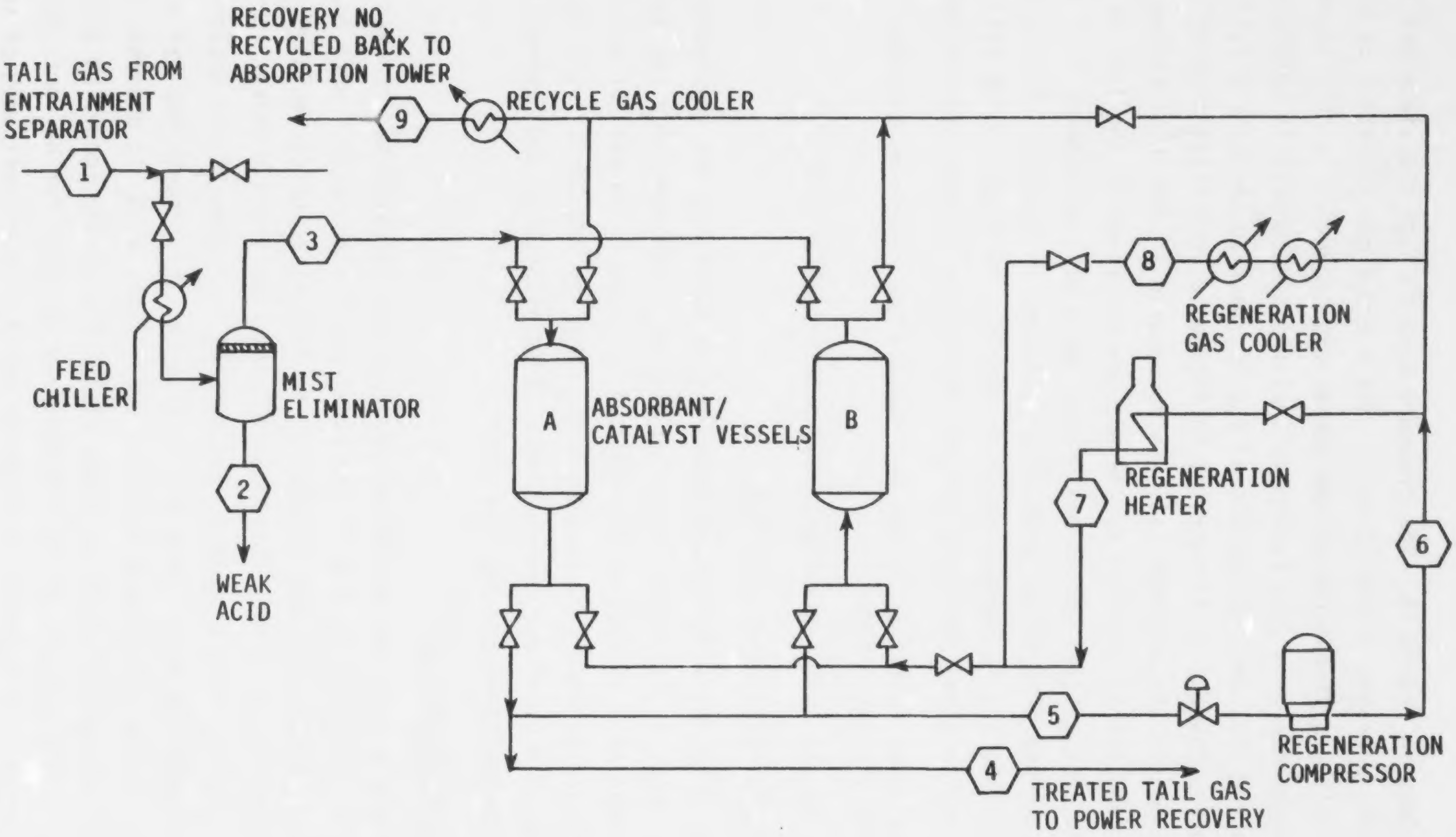
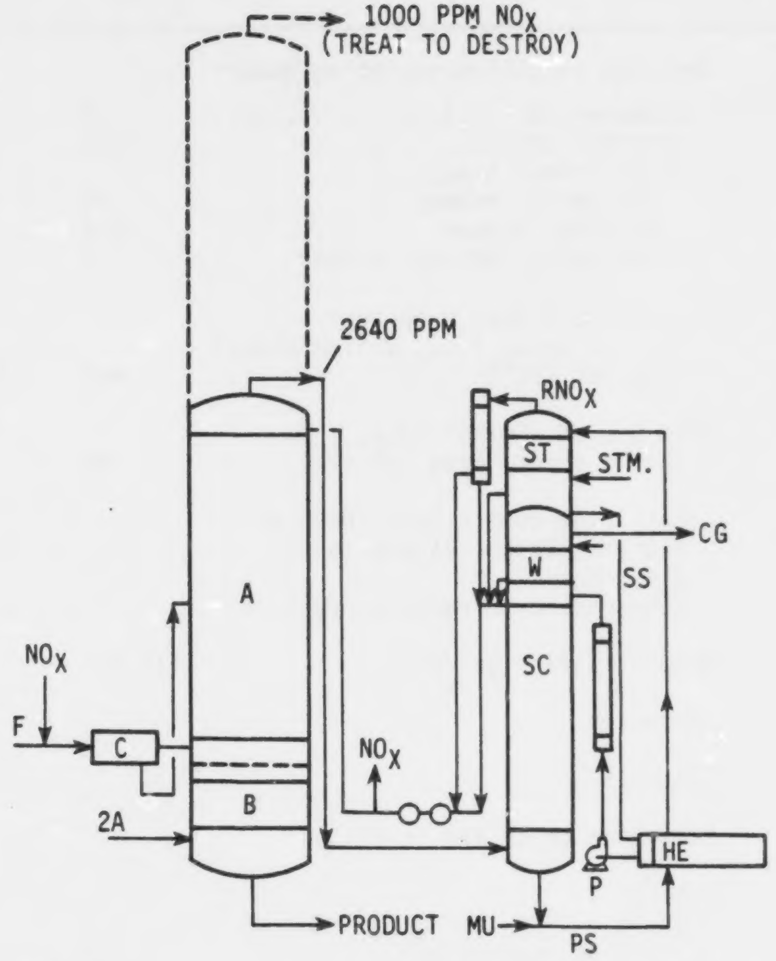

TAIL GAS FROM
ENTRAINMENT
SEPARATOR
1
RECOVERY NO$_x$
RECYCLED BACK TO
ABSORPTION TOWER
9
RECYCLE GAS COOLER
3
FEED
CHILLER
MIST
ELIMINATOR
2
WEAK
ACID
A
ABSORBANT/
CATALYST VESSELS
B
8
REGENERATION
GAS COOLER
REGENERATION
HEATER
7
6
5
REGENERATION
COMPRESSOR
4
TREATED TAIL GAS
TO POWER RECOVERY

Figure 16. Two-bed Purasiv N process (Vessel A under adsorption and Vessel B under regeneration heating) for control of NO_x in nitric acid plant tail gas.[19]


1000 PPM NO$_X$
(TREAT TO DESTROY)
2640 PPM
RNO$_X$
ST
STM.
CG
W
SS
A
SC
NO$_X$
F
C
NO$_X$
B
2A
HE
P
PRODUCT
MU
PS

F = FEED

C = CONDENSER

B = BLEACHING SECTION

A = ABSORBER

SC = SCRUBBING SECTION

W = WASHING SECTION

CG = CLEAR GAS

FW = FEED WATER

AW = ACID WASH

PS = PREGNANT SCRUBBING SOLUTION

HE = HEAT EXCHANGER

ST = STRIPPING TOWER

SS = STRIPPED SOLUTION

T-C = TRIM COOLER

RNO$_X$ = RECOVERED NITRIC OXIDER

BL = BLEED

RA = RECOVERED ACID

Figure 17. The Bolme NO$_x$ recovery system.[23]

TABLE 8. DESIGN SPECIFICATIONS FOR THE BOLME NO_x RECOVERY PROCESS APPLIED TO A NEW 220-METRIC-TON-PER-DAY MEDIUM-PRESSURE NITRIC ACID PLANT[23]

Combined scrubbing-stripping tower:		
Diameter, m (in.)	2	(80)
Internal cooling	none	
Trays (sieve type)		
Scrubbing, number	15	
Washing, number	3-4	
Stripping section, number	3	
Feed/Product heat exchanger (Spiral type, i.e., coiled sheet) area, m^2 (ft^2)	440	(4,750)
Trim Cooler (Spiral type, i.e., coiled sheet) area, m^2 (ft^2)	65	(700)
Liquid ring compressors (Nash or Siemens-Hinsch) Vacuum-to-atmospheric Atmospheric-to-recycle pressure		
Pump, liter/h (gal/min)	115,000	(500)
Condenser		
Controls		
Operating costs and benefits		
Utilities:		
Steam @ 5 $lb/in.^2$ gauge (130 kPa·gauge), kg/h (lb/h	1,745	(3,850)
Power, kW	125	
Cooling water, liter/min (gal/min)	2,500	(660)
Benefits:		
HNO_2 from RNO_2 (2500 → 100 parts/million), kg/day (lb/day)	4,670	(10,295)

year. As indicated in Figure 17, one major advantage of the process is that the nitric acid plant absorber is 40 percent smaller than one required to reduce the NO_x tail gas concentration to 1000 ppm. Another significant operating advantage of the process is that it tends to compensate for upsets. For example, a 2640 ppm jump in the NO_x entering the recovery process will produce only a 283 ppm jump in the NO_x in the exhaust.

The Bolme process is based on the same three reactions as the nitric acid process itself:

1. $2NO + O_2 \rightarrow 2NO_2$
2. $2NO_2 + H_2O \rightleftarrows HNO_2 + HNO_3$
3. $3HNO_2 \rightleftarrows HNO_3 + H_2O + 2NO$

The scrubbing solution employed in the process stabilizes nitrous acid and removes it as fast as it is formed. This is accomplished by using a 25 to 30 weight percent nitric acid solution that can reverse the last reaction, but allows the second reaction to proceed in the indicated direction. The solubility of nitrous acid is maximum in this concentration of nitric acid. The recovery process chemistry is complimentary to that of the basic nitric acid process. At low NO_x partial pressures, the Bolme process works well, while at high NO_x partial pressures, the basic nitric acid process works efficiently.

As illustrated in Figure 17, the integrated NO_x control method begins with the normal nitric acid process. The feed (F) passes through a condenser (C) and enters the absorption tower. Secondary air passes through a bleaching section (B) and joins the feed. These combined streams then enter the plant absorber (A); tail gas from the absorber contains 2600 ppm NO_x. Tail gas is then directed to the scrubbing section (Sc) where the NO_x content is reduced to approximately 150 ppm (70% NO_2). Exit gas from the scrubber enters the washing section (W) where it is contacted with feed water (FW) to convert NO_2 to HNO_3 and NO. Clean gas (CG) from the washing section is sent to the power recovery and the acid wash (AW) is fed to the absorber. Pregnant scrubbing solution (PS) is heated to approximately 70°C in a feed-product heat exchanger (HE) before it is stripped with steam in the stripping tower (ST) to produce the stripped solution (SS). Stripped solution is first cooled in the same heat exchanger (HE) and then further cooled in the trim cooler (T-C) prior to recycle. Recovered nitrogen oxides (RNO_x) are cooled in a condenser and compressed for recycle; bleed (B1) is also compressed and the combined streams are recycled to the absorber inlet. Recovered acid (RA) joins the acid wash as feed to the absorber.

SECTION 4

STARTUP, SHUTDOWN, AND MALFUNCTIONS

Prior to the inspection of a nitric acid plant, the field inspector should become familiar with the plant's startup and shutdown procedures and also with possible plant malfunctions.

All ammonia oxidation nitric acid plants use platinum alloy catalysts to promote the oxidation reaction. The efficient life of a catalyst varies in each plant as a result of the plant's design and operation practices. In general, however, the catalyst at pressure process nitric acid plants is changed once a month. This catalyst renewal requires a complete plant shutdown and startup. Additional unplanned shutdowns and startups are due to abnormal process conditions such as abnormally high temperatures and/or pressures that jeopardize the mechanical or process equipment.

In addition to abnormal process shutdowns, any of the following service upsets or failures will result in a plant shutdown:

1. Mechanical and/or process equipment,
2. Ammonia supply,
3. Lack of usage or storage for nitric acid,
4. Waste heat boiler feed water,
5. Absorber feed water,
6. Cooling water,
7. Instrument air, and
8. Electric power.

The average modern nitric acid plant will have 20 to 30 set points that, when exceeded, will trigger an automatic plant shutdown. Shutdowns due to service failures and abnormal process conditions are a function of the owner's mode of performance. Poor maintenance and operation will result in frequent shutdowns. Good maintenance and operating habits, however, should minimize these shutdowns to approximately one per month.

4.1 STARTUP

Before the ammonia oxidation reaction is started in a nitric acid plant, not only must all of the process equipment be in operating condition, but all of the numerous services must be available. With these prestartup conditions assured, the next procedures are:

1. The air compressor has been run at a low rate until it is at its operating temperature and pressure.
2. The ammonia vaporizing and supervising system is at operating temperature and pressure.
3. All downstream equipment such as heat exchangers, waste heat boilers, and cooler-condenser are warmed up with auxiliary steam.
4. The cooling water is circulating in the absorber.
5. The absorber feed water rate has been adjusted for startup.
6. The platinum catalyst gauze has been preheated and ready for the light-off.

Under these conditions, the air flow is diverted to the ammonia-air mixer and the ammonia vapor is added. The mixture passes over the catalyst in the converter, which is at ignition temperature, and the ammonia oxidation reaction starts. As the hot reaction gases pass through the preheated exchanger train, the warmup stream is shut off and a water spray wash is turned on to wash out ammonium nitrate and nitrite salts that form during this period of the startup (these salts are formed by unreacted ammonia and nitrogen oxides fumes during startup and are highly explosive). The bleach air flow is started and all air and ammonia flows are increased and adjusted until the desired operating conditions and rates are reached. The reaction gases flow to the absorber where the oxidation of nitrogen oxide continues and absorption produces the product acid.

The tail gas leaving the absorber reaches its equilibrium when the process upstream has leveled out at the selected operating rate temperature, pressure, etc. Only when this is achieved and the reheated tail gas is driving the power recovery expander can a catalytic reduction unit be put on stream. If the reduction unit were placed on stream before the tail gas had reached its reheated temperature, reaction ignition would not take place and the unit would not operate. The tail gas composition must be stabilized with the oxygen concentration design limits when the unit is ignited and put in operation because wide oxygen fluctuations would prevent proper operation. Concentrations above the design concentrations will produce temperatures above the thermal limitation of the catalyst and result in a safety shutdown. During this period before the catalytic reduction unit can be put on stream, very careful startup operation is necessary to keep the plant emissions to a minimum.

Should the plant be designed with sufficient absorption efficiency and no control device, the startup must be well controlled to keep the absorption process stabilized. With the absorption process under control, no difficulty should be experienced in meeting the standard during the startup period.

Desirable conditions for adsorption systems (molecular sieves) are constant bed temperatures, feed rates, and gas composition. If a nitric acid plant is equipped with an adsorption system, it must be preheated and put on stream when the tail gas composition and temperature have stabilized. This means that the unit will be the last equipment put into operation. As with a catalytic reduction system, before the adsorption unit is in operation, very careful startup operational procedures are required to keep the plant's emissions to a minimum.

4.2 SHUTDOWN

The first step in the shutdown of a nitric acid plant is to reduce the production rate, usually to approximately one-half of the design rate. This is done by reducing the reaction air flow to the ammonia-air mixer. The flow ratio controller maintains the correct amount of ammonia flow as the air flow is reduced. Next, the ratio of ammonia to ammonia-air mixture is reduced, which lowers the reaction temperature (catalyst gauze); when the catalyst has cooled to approximately 1000°F, the ammonia flow is shut off. After the ammonia flow is shut off, the catalytic unit should be shut down because the oxygen concentration in the tail gas will increase and the catalytic unit temperature will rise to the thermal limitation set point and it will cause an automatic shutdown. At this point, the air flow is further reduced; care must be taken to maintain the pressure of the absorption column, or excessive emissions will result. The air flow is continued until all nitrogen oxides are purged from the tail gas heater, waste heat boiler, and cooler-condensers. All drains from these exchangers are opened to purge condensed water and weak acid. The bleach air is now shut off and the absorption water to the absorber is reduced. The air to the ammonia-air mixer may now be reduced to a minimum permitting the compression train to turn slowly and cool. The compression train must be cooled down carefully to avoid damage. As the plant is cooled down, all services such as boiler feed water, absorber water, and cooling water should be shut off. During this purge and cooling of the plant, the absorber pressure and cooling must be maintained until the maximum amount of unabsorbed nitrogen oxides are absorbed, or excessive emissions will result.

A nitric acid plant designed with sufficient absorption capacity without an emission control system will have the same shutdown procedure as described in the previous paragraph (excluding the catalytic unit shutdown). The same care must be exercised to maintain absorption pressure and cooling or excessive emissions will result.

If a nitric acid plant is equipped with an adsorption system, it must be purged back into the absorber (all beds) to remove all nitrogen oxides before the ammonia feed to the converter is shut off. This cleans the unit of nitrogen oxides. The nitrogen oxides are absorbed before the plant temperatures change and while the purge gas is normal. If the adsorption unit was operated to the end, the oxides would have to remain in the cold adsorption beds or be purged to the atmosphere. It would not be desirable to keep the oxides absorbed in a cold adsorption bed. As in all nitric acid plants, during shutdown the pressure and cooling must be maintained on the absorber tower or excessive emissions will result.

4.3 MALFUNCTIONS

Any nitric acid plant designed with sufficient absorption capacity or equipped with emission control devices will have difficulty controlling their emissions if they experience any of the following upsets or malfunctions:

1. Insufficient process air (converter and bleach air) will result in an excess of unoxidized nitrogen oxide that the absorber and catalytic unit cannot eliminate.

2. Insufficient cooling of the cooler-condensers and absorber will hinder the absorption of nitrogen dioxide and tetroxide.

3. Operation of the cooler-condenser and absorber below the design pressure will lower the absorption efficiency and overload the abatement system with nitrogen oxides.

4. If the plant is operated above design production rate and/or acid concentration, the cooler-condensers and absorber will not perform at their designed efficiency.

5. The mechanical failure of pumps, control valves, instruments, etc., will create unstable operations such as insufficient cooling, poor reactant flow control, etc., which will hinder the proper absorption and catalytic unit operations.

6. Process equipment malfunctions such as leaking exchangers could easily allow bypassing of nitrogen oxides past the catalytic unit to the atmosphere. Plugged trays in the absorber would reduce absorption efficiency.

7. The loss of any utility such as waste heat boiler feed water, instrument air, electricity, and process water would trigger an emergency shutdown that would vent excessive nitrogen oxides to the atmosphere.

SECTION 5

PROCESS CONTROL SYSTEM AND EMISSION MONITORING INSTRUMENTATION

Monitoring instrumentation plays three very important roles in the operation of a nitric acid plant:

1. Informing the plant operators and inspectors of the various flows, temperatures, pressures, and concentrations necessary to define plant performance and emission control performance.
2. Performing process control functions such as automatic flow control, automatic temperature control, and other control functions that may be performed along with the monitoring of these functions.
3. Helping to protect the process from damaging and hazardous operating conditions through the use of alarms, which notify the operator when control limits are reached, and the use of trip-out and interlock systems, which shut down parts or all of the plant when certain preset control limits are reached.

Therefore, based on the above instrumentation functions, all nitric acid plant inspectors must be thoroughly familiar with all types of plants, control systems, and emission monitoring instrumentation that can be encountered in the industry. The following sections will define the general concepts of performing the above monitoring functions. After understanding the general concepts, the plant inspector must become familiar with the particular details of each plant's monitoring system. With a thorough understanding of the monitoring information, a plant inspector will be able to determine such important parameters as plant process and production rates during the inspection.

5.1 PROCESS MONITORING INSTRUMENTATION

Although the nitric acid manufacturing process has many variations, the basic ammonia-oxidation process can be considered a baseline for monitoring nitric acid manufacturing processes. Figure 18 is an instrumentation flow diagram of the AOP process.

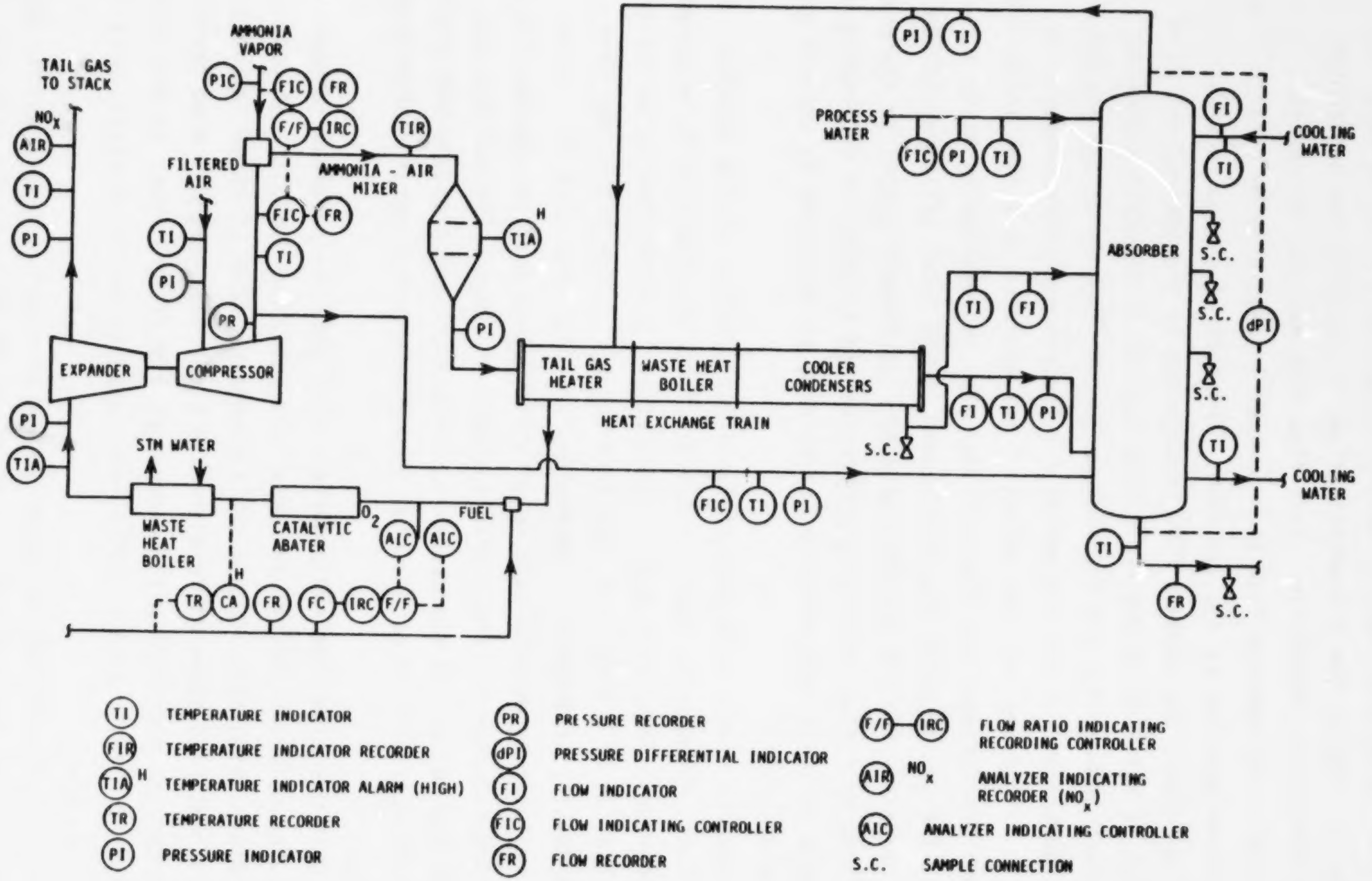

TAIL GAS TO STACK
NO_x AIR
TI
PI
AMMONIA VAPOR
PIC
FIC
FR
F/F
IRC
TIR
FILTERED AIR
AMMONIA - AIR MIXER
FIC
FR
TI
PI
TI
PR
TIA H
PI
EXPANDER
COMPRESSOR
PI
TIA
STM WATER
WASTE HEAT BOILER
CATALYTIC ABATER
O_2
AIC
AIC
FUEL
TR
CA H
FR
FC
IRC
F/F
TAIL GAS HEATER
WASTE HEAT BOILER
COOLER CONDENSERS
HEAT EXCHANGE TRAIN
S.C.
FIC
TI
PI
PI
TI
PROCESS WATER
FIC
PI
TI
TI
FI
FI
TI
PI
ABSORBER
FI
TI
COOLING WATER
S.C.
S.C.
S.C.
dPI
TI
COOLING WATER
TI
FR
S.C.
TI TEMPERATURE INDICATOR
TIR TEMPERATURE INDICATOR RECORDER
TIA H TEMPERATURE INDICATOR ALARM (HIGH)
TR TEMPERATURE RECORDER
PI PRESSURE INDICATOR
PR PRESSURE RECORDER
dPI PRESSURE DIFFERENTIAL INDICATOR
FI FLOW INDICATOR
FIC FLOW INDICATING CONTROLLER
FR FLOW RECORDER
F/F—IRC FLOW RATIO INDICATING RECORDING CONTROLLER
AIR NO_x ANALYZER INDICATING RECORDER (NO_x)
AIC ANALYZER INDICATING CONTROLLER
S.C. SAMPLE CONNECTION

Figure 18. Instrumentation flow diagram for ammonia oxidation process.

The plant production rate is controlled by the air and ammonia feed rate. The air rate to the air-ammonia mixer is manually set and controlled by a flow indicating controller. This flow rate may also be recorded by a flow recorder. The ammonia flow to the mixer is regulated by a ratio flow control system governed by the air flow. The control system records air and ammonia flow rates; the ammonia flow is adjusted to the desired set point ratio which is selected on the ratio flow control to maintain a constant concentration between 9.5 and 10.5 volume percent ammonia in the process stream. The balance of the air needed for the process (approximately 20 percent) is introduced into the bottom of the absorber by a flow indicating controller. This bleach air flow rate may also be recorded by using a flow recorder. The sum of these two air streams is the total air flow introduced into the process. The introduction of water (condensate grade) to the absorber is controlled by a flow indicating controller and is adjusted to produce the amount (at a definite concentration) of acid to be generated by the air and ammonia feed rates.

The rate of nitric acid product leaving the bottom of the absorber (or bleach column if the plant has one separate from the absorber) is measured by a flow recorder. The nitric acid concentration is determined by taking the specific gravity of a sample at regular intervals. Some plants may have a recording density instrument to determine the acid concentration. In any event, a reliable production rate measurement method must be provided and be sufficiently accurate to comply with the NSPS standard. The absorber gas flow inlet and outlet are equipped with temperature and pressure indicators that aid the operators in maintaining the optimum conditions for absorption efficiency.

The exit gas flow from the absorber (tail gas) has numerous temperature and pressure indicators before and after the equipment that it must pass through before being exhausted to the atmosphere. Some of these temperature indicators have high temperature alarms to alert the operators when there is danger to certain equipment with temperature limitations, such as the expander (temperature limit is usually 1250^{0}F) and catalyst in the catalytic unit (if the plant has one).

The catalytic unit may be controlled by an oxygen analyzer which, through a ratio controller, adjusts the fuel feed rate to the unit for efficient automatic operation.

The tail gas stream is analyzed for nitrogen oxides before being exhausted to the atmosphere. This instrument is required by the NSPS and must be an indicating-recording analyzer (reading in ppm nitrogen dioxide). It must be in continuous operation and maintained in optimum operating condition at all times.

The instrumentation shown in Figure 18 is only the minimum required for the process control. The following auxiliaries have numerous instruments and controls for their proper operation and plant protection, and the inspector should become familiar with instrumentation for these processes.

1. Compression train (startup turbine, compressor, and expander),
2. Ammonia vapor supply system,
3. Waste heat boiler feed water supply system,
4. Cooling water system,
5. Absorber water supply and treatment (condensate quality for absorption),
6. Product nitric acid storage, and
7. Electric supply system.

These various systems are interlocked with the process controls, so it is important that their possible effects on plant production and emission control be understood.

5.2 EMISSION CONTROL INSTRUMENTATION

It should be emphasized that there are many possible instrumentation concepts for the control techniques discussed in Section 3.3. The following section discusses a general type of instrumentation scheme for each control technique. The inspector should become familiar with the details of a given emission control instrumentation scheme prior to the inspection.

5.2.1 Catalytic Reduction

The control instrumentation used on catalytic reduction units is as varied as the number of suppliers. The simplified general flow diagram in Figure 19 illustrates the instrumentation scheme recommended by one supplier but is not the only system in use.

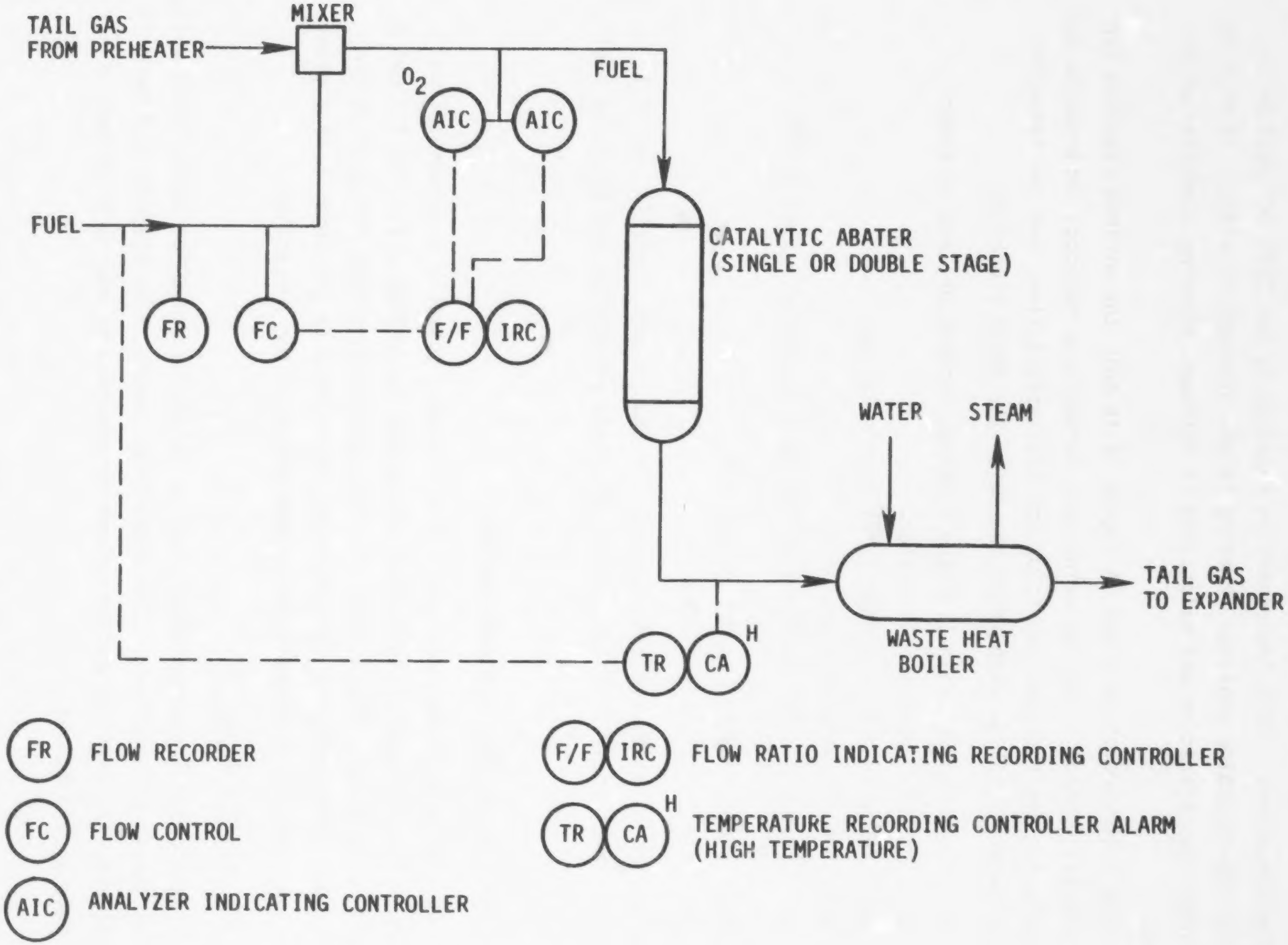

TAIL GAS
FROM PREHEATER
MIXER
FUEL
O2
AIC
AIC
FUEL
FR
FC
F/F
IRC
CATALYTIC ABATER
(SINGLE OR DOUBLE STAGE)
WATER
STEAM
TAIL GAS
TO EXPANDER
TR
CA
H
WASTE HEAT
BOILER
FR FLOW RECORDER
FC FLOW CONTROL
AIC ANALYZER INDICATING CONTROLLER
F/F IRC FLOW RATIO INDICATING RECORDING CONTROLLER
TR CA H TEMPERATURE RECORDING CONTROLLER ALARM
(HIGH TEMPERATURE)

Figure 19. Catalytic reduction unit instrumentation flow diagram.

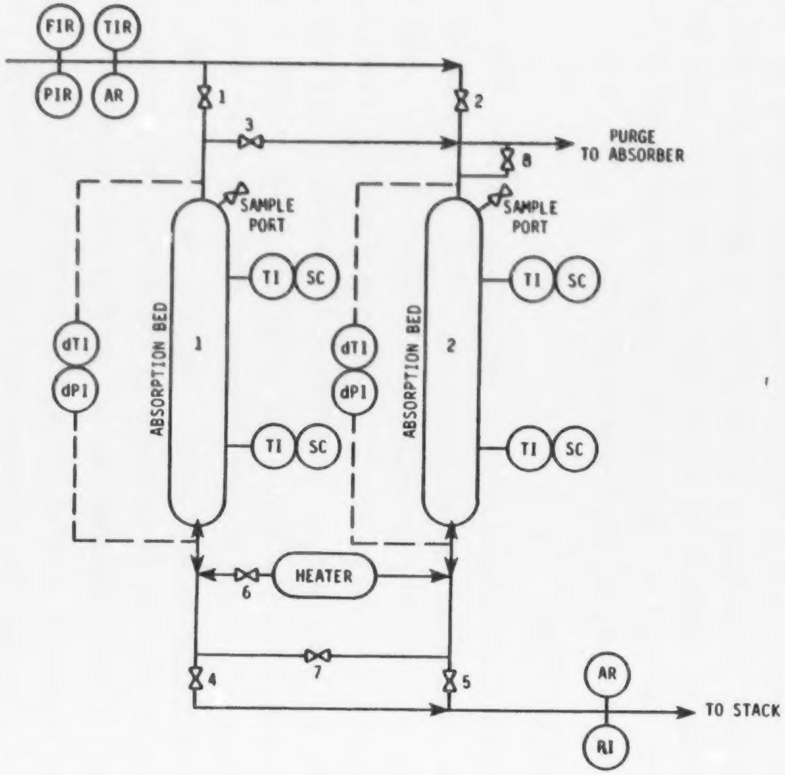
The fuel must be clean (free of sulfur compounds, etc.) to protect the catalyst and should be well mixed with the tail gas before passing over the catalyst. The flow rate of fuel gas is controlled by the oxygen concentration in the tail gas. The oxygen concentration is determined by a continuous analyzer that measures and controls the fuel flow rate. The fuel concentration in the tail gas is analyzed by an analyzer-controller that maintains the flow rate as required by the oxygen analyzer and controller. The ratio of fuel to oxygen is controlled by a ratio flow controller in the system. This ratio is selected to maintain optimum operation and protect the catalytic unit from excess fuel that would decompose and poison the catalyst (carbon). In addition, a temperature recording controller with a high alarm is used to warn the operator of abnormally high temperatures (usually about 1500°F) that would damage the catalyst. As an additional safety precaution, this control will also shut off the fuel supply if there is a lapse in time.

5.2.2 Absorption

Emission control by absorption is somewhat misleading as no emission control equipment is necessary if the plant is designed and built with sufficient absorption capacity to ensure that the emissions will be below the NSPS standard. A plant so designed will need no additional instrumentation because the normal process instrumentation will be sufficient.

5.2.3 Molecular Sieves

The operation of molecular sieve adsorption systems tends to be simple. Most operate on automatic valve sequencing, with time mechanisms providing primary control. Performance evaluation is based on flow, temperature, pressure, and composition measurements. The adsorption instrumentation diagram shown in Figure 20 schematically illustrates desirable instrumentation. The analyzer-recorders for the inlet and outlet gas flows determine the nitrogen oxides concentrations as nitrogen dioxide. From these two analyzers, the unit's efficiency can be monitored. The exit analyzer does not replace the continuous stack monitor as required by the NSPS. The various sample connections are not only designed for gas samples but for absorbent samples as well. These absorbent samples are useful in measuring the unit's performance and in projecting when the absorbent will have to be changed. These adsorbent analyses also help to identify other adsorbates and adsorbent poisons.


FIR
TIR
PIR
AR
1
2
3
8
PURGE TO ABSORBER
SAMPLE PORT
SAMPLE PORT
TI
SC
TI
SC
TI
SC
TI
SC
dTI
dPI
dTI
dPI
ABSORPTION BED
1
ABSORPTION BED
2
HEATER
6
7
4
5
AR
RI
TO STACK

VALVE SEQUENCE

NO. 1 IN USE		NO. 2 IN USE	
VALVE NO.	POSITION	VALVE NO.	POSITION
1	OPEN	1	CLOSED
2	CLOSED	2	OPEN
3	CLOSED	3	OPEN
4	OPEN	4	CLOSED
5	CLOSED	5	OPEN
6	OPEN	6	OPEN
7	OPEN	7	OPEN*
8	OPEN	8	CLOSED

*NO. 7 OPENED AFTER CLOSING NO. 6

INSTRUMENTS

FIR	FLOW INDICATING RECORDER
TIR	TEMPERATURE INDICATING RECORDER
PIR	PRESSURE INDICATING RECORDER
AR	ANALYZER RECORDER
TI	TEMPERATURE INDICATOR
SC	SAMPLE CONNECTION
PI	PRESSURE INDICATOR
dTI	TEMPERATURE DIFFERENTIAL INDICATOR
dPI	PRESSURE DIFFERENTIAL INDICATOR

Figure 20. Molecular sieve adsorption unit instrumentation flow diagram.

5.3 EMISSION MONITORING INSTRUMENTATION

Several different types of continuous emission monitoring techniques may be used to determine the NO_x concentrations in the tail gas prior to release. Some of these methodologies are summarized below. Each system has both advantages and disadvantages and the monitor selector must take this into consideration along with the stack conditions expected to be encountered.

5.3.1 Ultraviolet Absorption

The main type of ultraviolet absorption monitor is the nondispersive ultraviolet analyzer shown in Figure 21. This analyzer uses the concept of a split-beam photometer. It monitors the concentration of a pollutant by measuring the difference in the amount of radiation absorbed at two different wavelengths (ultraviolet, visible, or near-infrared). Radiation from the source, usually a gas discharge lamp, is partially absorbed as it passes through a sample of gas. As radiation leaves the sample, it is divided into two beams by a semitransparent mirror. Each beam then passes through an optical filter that removes all wavelengths except the one to be measured. The filtered beam then strikes a phototube. The beam splitter is set up to reflect and transmit so that the intensities of the radiation striking each phototube will be nearly equal during normal operation of the analyzer.

The beam reaching one phototube is in a wavelength that is absorbed strongly by the pollutant being measured; thus, this beam actually measures the concentration. The beam directed to the second phototube is in a wavelength that is absorbed weakly or not at all by the pollutant and is used as a reference. If the concentration of the absorbing pollutant changes, the intensity of radiation reaching the first phototube varies in accordance with the concentration of pollutant. The radiation to the second phototube varies very little, or not at all.

In each phototube, the current flow is proportional to the intensity of radiation striking the phototube. This current is fed to an amplifier that outputs a dc voltage that varies logarithmically with the current, and, hence, with the intensity of the radiation. Voltage output from the reference beam amplifier is subtracted from that of the measuring beam amplifier. This difference in voltage ouput varies linearly with the concentration of the pollutant being measured. Therefore, the gaseous pollutant concentration may be read directly or indirectly through the difference in output voltage.

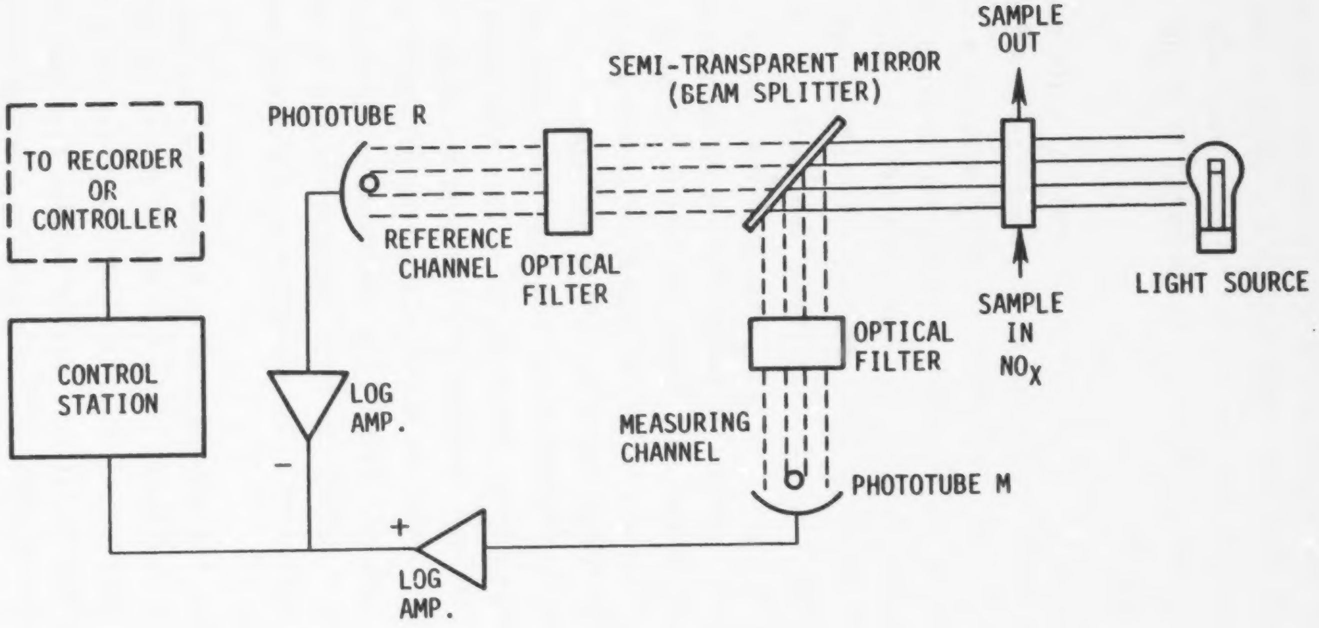

SAMPLE OUT
SEMI-TRANSPARENT MIRROR (BEAM SPLITTER)
PHOTOTUBE R
TO RECORDER OR CONTROLLER
REFERENCE CHANNEL
OPTICAL FILTER
LIGHT SOURCE
SAMPLE IN NO_X
OPTICAL FILTER
CONTROL STATION
LOG AMP.
−
MEASURING CHANNEL
PHOTOTUBE M
+
LOG AMP.

Figure 21. Nondispersive ultraviolet analyzer.

5.3.2 Infrared Absorption

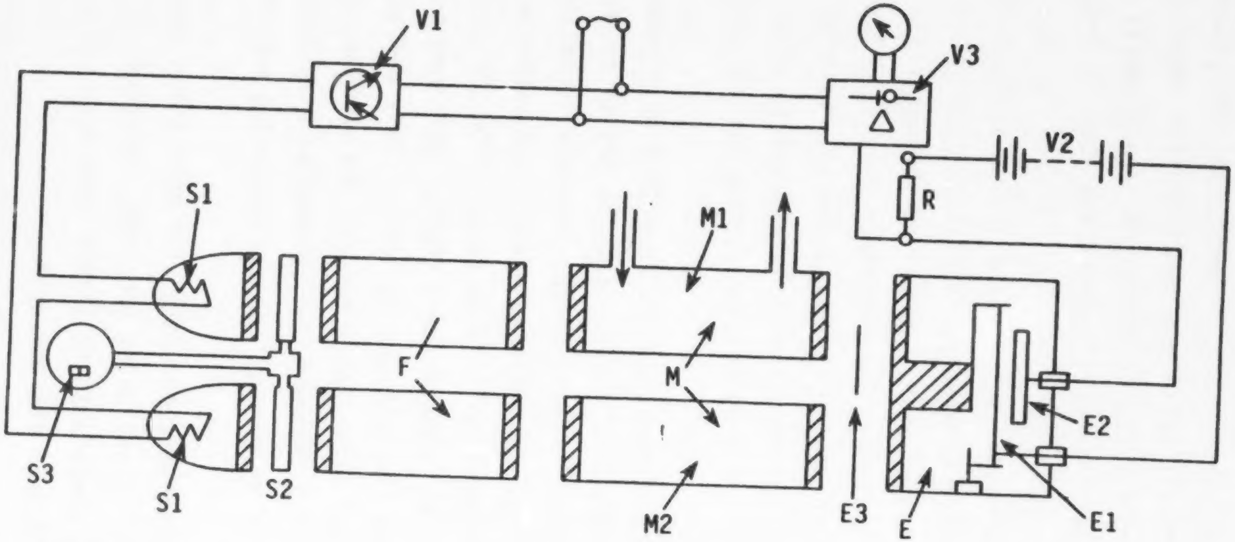
Two main types of infrared sensors are commonly used--nondispersive and dispersive. The term "nondispersive" refers to the lack of a light-dispersing element such as a prism or grating that selects a particular analytical wavelength of light. The more traditional Luft-type nondispersive infrared (NDIR) analyzer has been used to make process and environmental measurements of nitric oxide for over 20 years. A schematic of this type of analyzer is shown in Figure 22.

In the NDIR analyzer, two identical infrared-radiation emitters serve as matched sources of broad-brand infrared energy. Radiation from these sources is modulated by a motor-driven chopper disk, and passed through filters and measuring cells into an energy receiver. The reference cell is filled with a gas such as nitrogen that does not absorb infrared energy. Another cell contains the gas being analyzed. The amount of infrared energy passing through the analysis cell that will be absorbed by the gas depends on the concentration and wavelength band of the gas.

The energy receiver consists of two chambers separated by a membrane. Both chambers are filled with a mixture of argon and the gas to be measured. As the gas absorbs infrared energy, it heats up and pressure increases. If the two chambers are exposed to equal amounts of energy, the membrane will remain in a neutral position and there will be no output from the instrument. Because the gas in the analysis cell absorbs more energy, pressure on that side of the chamber increases and the membrane deflects. This deflection is detected as a change in electrical capacitance between the membrane and a fixed electrode and therefore, as a change in ac voltage at a resistor. This ac voltage change is displayed as dc current using a measuring amplifier on an indicating instrument.

If the gas to be tested contains components with absorption bands that slightly overlap those of the pollutant being measured, identical filter cells are filled with the interfering components to screen them out. This "positive filtering" makes it easier to measure the pollutant.

The Luft-type NDIR analyzer generally uses an extractive sampling system to transport the sample of stack gas to the analyzer. Particulate matter and water vapor that interfere with the species measurement are removed. Generally, Luft-NDIR analyzers are used to monitor SO_2, NO_2, NO, CO, and CO_2.



E - ENERGY RECEIVER
E1 - MEMBRANE
E2 - ELECTRODE
E3 - RECEIVER TRIMMER
F - FILTER CELLS

M - MEASURING CELLS
M1 - ANALYSIS CELL
M2 - REFERENCE CELL
R - HIGH-IMPEDANCE RESISTOR
S1 - RADIATION SOURCES

S2 - CHOPPER DISK
S3 - CHOPPER MOTOR
V1 - POWER SUPPLY
V2 - DC VOLTAGE SOURCE
V3 - AMPLIFIER

Figure 22. Nondispersive infrared analyzer.

A schematic of another nondispersive analyzer is shown in Figure 23. In contrast to the Luft-type analyzer, which looks at a broad spectral region and must be sensitized for each particular gas using a detector cell, this NDIR spectrometer can be set at any wavelength within its range. In addition, the adjustable analyzer can measure any gas within its spectral region, and unlike the Luft-type analyzer, is not limited to a single, preselected gas. When many absorbing gases are present, however, it may be difficult to locate a wavelength in a spectral region where other gases do not absorb.

The analyzer shown in Figure 23 uses a narrow-band pass filter that scans the spectral region between 2.5 and 14 microns. Because the analyzer has a variable path length from 3/4 to 20 meters, it has a large sensitivity range. These and similar analyzers can measure many gases simultaneously; however, other gases, water vapor, or particulates may cause absorption problems in the spectral region of the pollutant.

Another type of NDIR analyzer is the gas cell correlation spectrometer shown in Figure 24. EPA researchers are investigating this type of analyzer as an in-situ analyzer for various pollutants. The analyzer uses a broad band IR source and detector, one or two cells filled with varying concentrations of the gas to be measured to provide the wavelength selection. This technique has shown potential for rejecting interference. Thus, it can be used to improve low level sensitivities, or to simplify or eliminate requirements for sampling systems. In addition, switching selection cells allows several pollutants to be monitored by the same unit. Figure 25 shows a single-beam, dual wavelength NDIR analyzer. In this analyzer, a single IR radiation beam is passed through the sample gas and filtered into two wavelengths by a chopper. The wavelengths are chosen so that one wavelength has no absorption, while the other has maximum absorption. The ratio of their absorptions is sensed by a conventional thermal electric IR detector.

5.4 CHEMILUMINESCENCE

Chemiluminescence is a chemical and optical monitoring technique. In this technique, a gas molecule reacts with a reagent to form an excited molecule that spontaneously decays, producing photoemissions. Sensitivity, rapid response time, and instrument stability make the chemiluminescence method

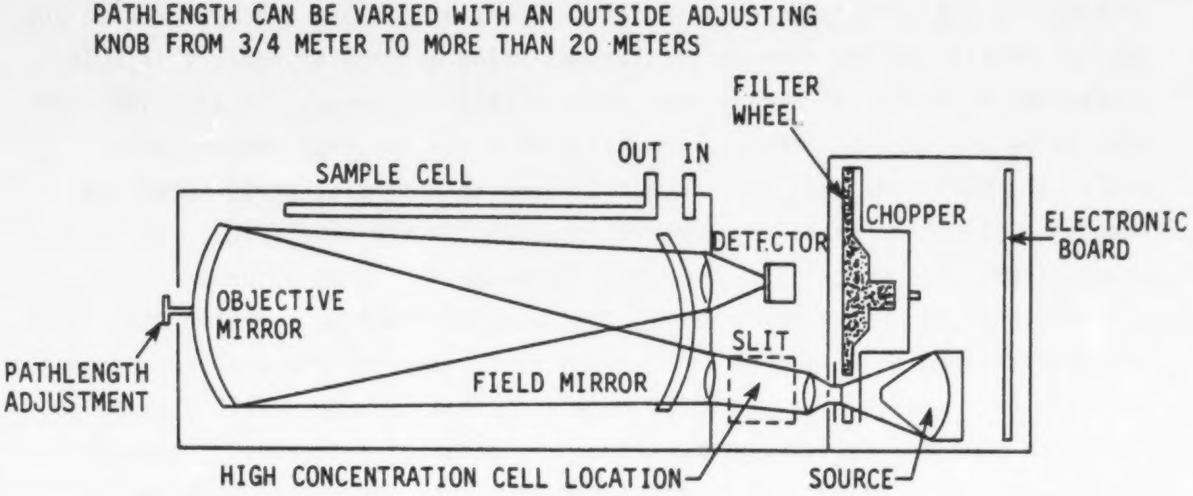

PATHLENGTH CAN BE VARIED WITH AN OUTSIDE ADJUSTING KNOB FROM 3/4 METER TO MORE THAN 20 METERS
FILTER WHEEL
OUT
IN
SAMPLE CELL
CHOPPER
ELECTRONIC BOARD
DETECTOR
OBJECTIVE MIRROR
PATHLENGTH ADJUSTMENT
SLIT
FIELD MIRROR
HIGH CONCENTRATION CELL LOCATION
SOURCE

Figure 23. Variable wavelength NDIR analyzer.

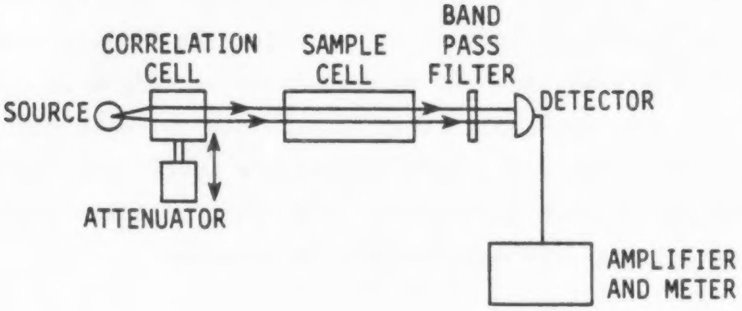

BAND PASS FILTER
CORRELATION CELL
SAMPLE CELL
SOURCE
DETECTOR
ATTENUATOR
AMPLIFIER AND METER

Figure 24. Gas cell correlation spectrometer analyzer.

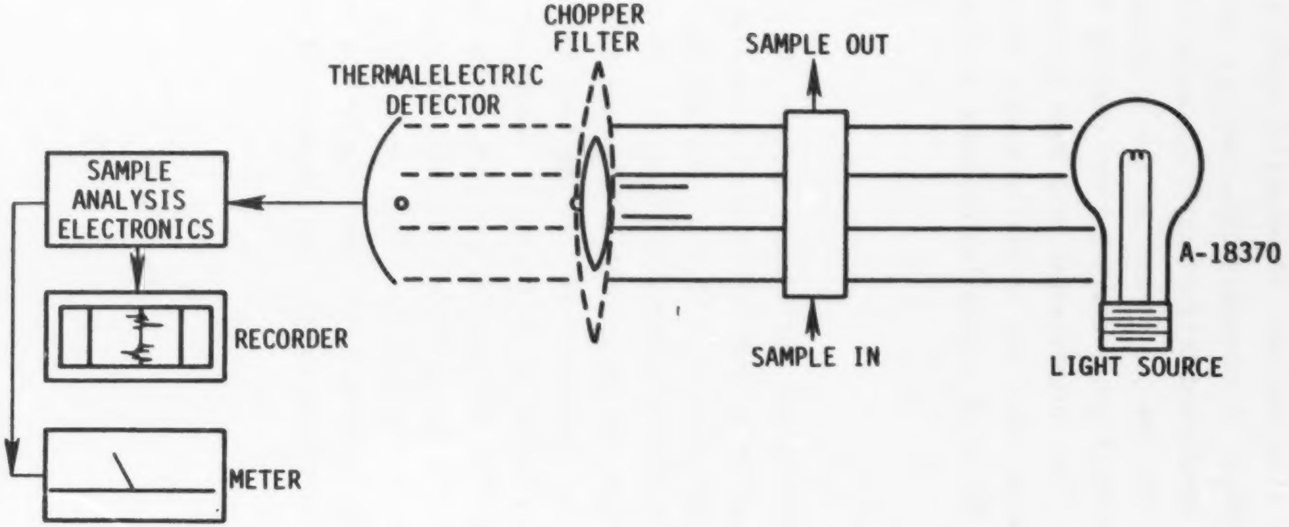

CHOPPER
FILTER
THERMALELECTRIC
DETECTOR
SAMPLE OUT
SAMPLE
ANALYSIS
ELECTRONICS
A-18370
RECORDER
SAMPLE IN
LIGHT SOURCE
METER

Figure 25. Single-beam, dual wavelength NDIR.

suitable for continuous monitoring, although care must be taken to maintain the monitored stream at constant flow rates. A typical NO and NO_x analyzer is shown in Figure 26. In this analyzer, NO molecules combine with O_3 to form an excited molecule (NO_2). A photomultiplier detects photoemission decay and sends the signal to a sample-and-hold circuit. To detect NO_2, a NO_2 and NO converter is connected into the gas stream and the NO analysis is run again. After the second pass, a circuit subtracts the first reading from the second to obtain the NO_2 reading. Few interferences have been observed, but high concentrations of CO_2 or water vapor may partially quench the chemiluminescence. In addition to monitoring NO and NO_x, chemiluminescence devices are also used to detect O_3 and SO_2.

5.5 ELECTROCHEMICAL

Analyzers using electrochemical transducers measure the current induced by the electrochemical oxidation of the pollutant at a sensing electrode. Sensors are available for measuring SO_2, CO, H_2S, NO, and NO_2. Figure 27 shows a simplified schematic of an electrochemical transducer. In this analyzer, the pollutant diffuses through the semipermeable membrane into the transducer at a rate proportional to the concentration. At the sensing electrode, the pollutant undergoes an electrochemical oxidation or reduction that causes a current directly proportional to the partial pressure of the gas being monitored. Since electrons are produced at the sensing electrode, this electrode is at a lower potential than the counter electrode. Thus, an electron current can flow from the sensing electrode through the amplifier to the counter electrode, and the current will be proportional to the concentration of the pollutant.

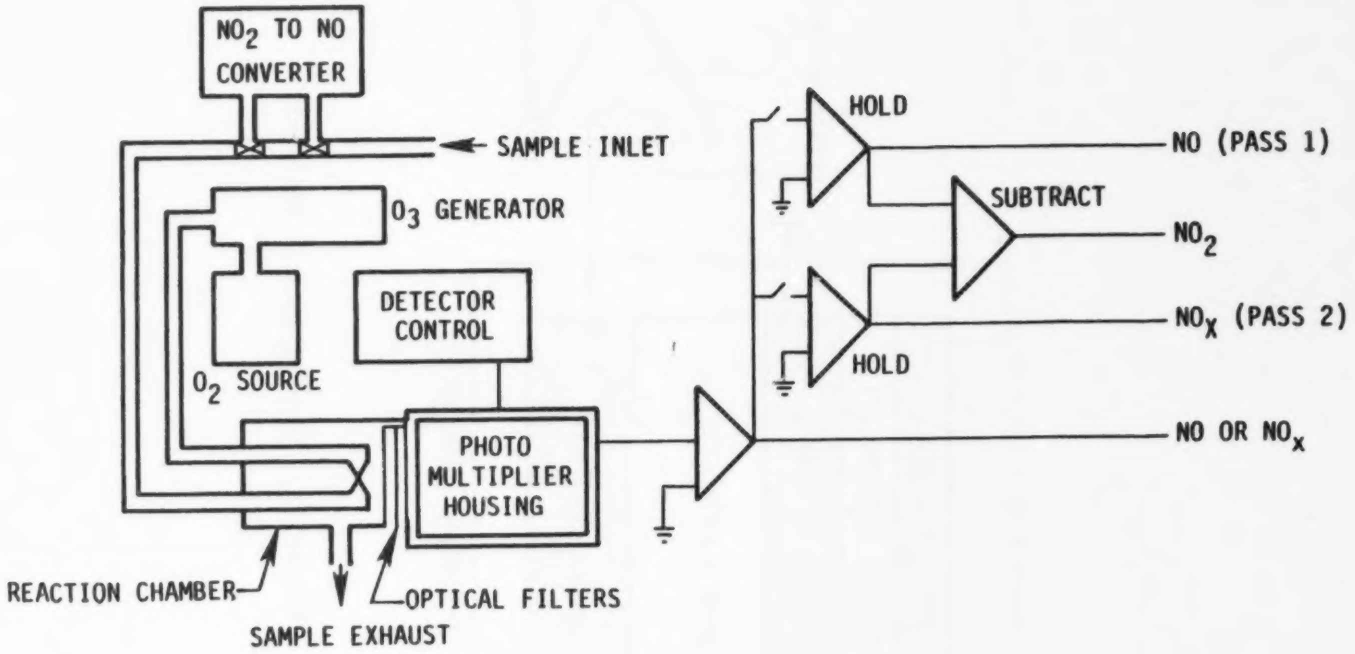

NO2 TO NO CONVERTER
SAMPLE INLET
O3 GENERATOR
O2 SOURCE
DETECTOR CONTROL
PHOTO MULTIPLIER HOUSING
REACTION CHAMBER
SAMPLE EXHAUST
OPTICAL FILTERS
HOLD
SUBTRACT
HOLD
NO (PASS 1)
NO2
NOX (PASS 2)
NO OR NOx

Figure 26. Chemiluminescence analyzer.

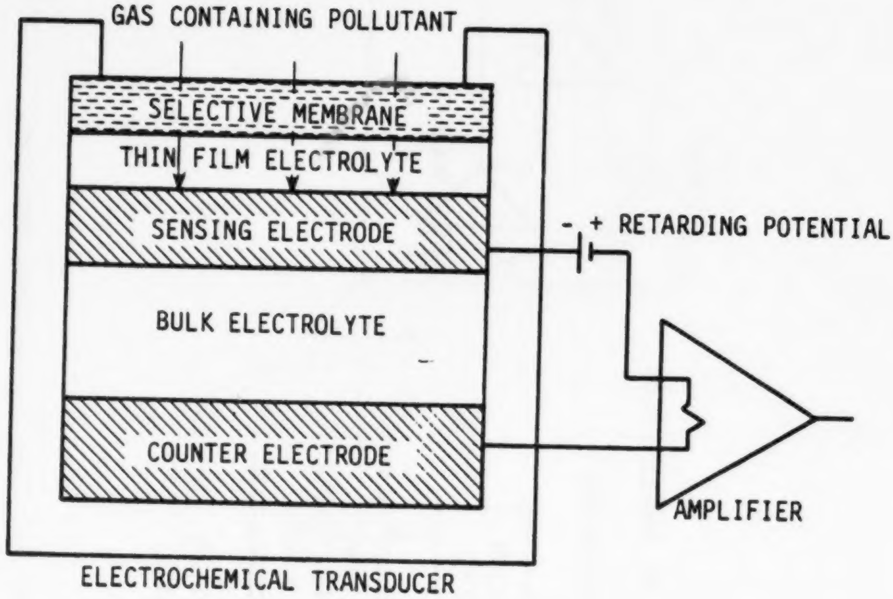

GAS CONTAINING POLLUTANT
SELECTIVE MEMBRANE
THIN FILM ELECTROLYTE
SENSING ELECTRODE
- + RETARDING POTENTIAL
BULK ELECTROLYTE
COUNTER ELECTRODE
AMPLIFIER
ELECTROCHEMICAL TRANSDUCER

Figure 27. Electrochemical analyzer.

SECTION 6

PERFORMANCE TESTS AND CONTINUOUS MONITOR PERFORMANCE EVALUATIONS

6.1 PERFORMANCE TESTS

Compliance with the emission limitations of the NSPS is demonstrated through performance tests conducted as required by Section 60.8. The initial performance tests are required within 60 days of achieving maximum production rate, but no later than 180 days after initial startup. The plant owner or operator is required under Section 60.8(d) to provide at least 30 days prior notice to the Administrator before conducting performance tests. In addition, the plant owner or operator must also notify the Administrator 30 days prior to the expected date of initial startup and within 15 days of actual startup that such is occurring.

The test methods used by nitric acid plants in demonstrating compliance are Method 7 for determination of NO_x concentration, Method 1 for sampling point determination, Method 2 for determination of velocity and volumetric flow rate, and Method 3 for gas analysis. A copy of these Methods is provided in Appendix B.

6.2 PRETEST ARRANGEMENTS

Field enforcement personnel should arrange a pretest meeting with responsible plant personnel to review the methods of testing and regulatory requirements. Although the requirements are specified in the NSPS, this meeting would clarify the testing requirements and avoid any potential misunderstanding associated with the testing requirements during onsite testing.

It is recommended that a pretest protocol be established that sets forth certain conditions such as production rate, acid strength, the sampling location(s), methods to be used, and date(s) of test(s). A pretest protocol will help to address any special circumstances that may arise by specifying procedures that will be used. The objective in establishing this protocol is to ensure that a representative performance test is conducted at conditions representative of the nitric acid plant's normal operation.

6.3 OBSERVING PERFORMANCE TESTS

Agency personnel observing performance tests should ensure themselves that the test is conducted under representative conditions. This does not require, however, that the agency representative observe the entire stack sampling procedure to determine if each run is conducted in accordance to the test method. Agency personnel should be more concerned with establishing that the nitric acid plant conditions are representative because the test methods are relatively rigid and the requirements straight forward.

The agency representative should observe, at the minimum, the following parameters at the nitric acid plant:

1. Ammonia feed rate,
2. Air flow rate to ammonia burner,
3. Catalyst temperature at ammonia burner,
4. Bleach air flow rate,
5. Ammonia burner and absorption column pressure,
6. NO_x concentration in tail gas,
7. Acid strength, and
8. Acid production rate (either at production strength or 100 percent basis). A material balance should be performed to confirm accuracy.

In addition to these parameters, the operating temperatures, pressures, flow rates, and any operating cycles associated with the control equipment should be noted. Although many of these values are not directly required for the calculation of emission rates, they are useful in establishing the representativeness of any future performance tests and in the evaluation of operating conditions in followup inspections.

6.4 CONTINUOUS MONITOR PERFORMANCE EVALUATION

Prior to conducting tests to determine compliance with the emission limitations, Section 60.13 requires that the continuous monitor be installed and operating. The monitor must have completed the 168-h conditioning period specified in Performance Specification 2, Appendix B of the NSPS. Section 60.13 also requires the continuous monitor to be certified through a performance evaluation, also in accordance with Performance Specification 2, within 30 days of any performance test required by Section 60.8; in addition, the Administrator must be furnished with two copies of the report within 60 days of the monitor evaluation.

Briefly, the continuous monitor performance evaluation consists of two portions: the conditioning period and the operating test period. The conditioning period is 168 hours of continuous normal operation where negative zero drift is quantified by offsetting the zero setting by 10 percent of the span. The operating test period is the portion of the monitor performance evaluation, 168 hours in duration, where the monitor is calibrated and checked for accuracy, drift, and response time.

The accuracy of the monitor is determined by comparing monitor output values with values obtained by the performance test reference methods. In addition, the monitor calibration error is checked by comparison of the monitor response with samples of known gas concentrations. Monitor drift is checked throughout the monitor test period as is monitor response time. The monitor requirements are summarized below.

6.4.1 Continuous Monitor Performance Specifications

1.	Accuracy (error)	≤20 percent of mean value of reference method test data (95 percent confidence interval)
2.	Calibration (error)	≤5 percent of each (50 percent, 90 percent) calibration gas mixture value
3.	Zero drift (2-h)	2 percent of span
4.	Zero drift (24-h)	2 percent of span
5.	Calibration drift (2-h)	2 percent of span

6.	Calibration drift (24-h)	2.5 percent of span
7.	Response time	15 min
8.	Operational period	168 h (minimum)

The span value for nitric acid plants subject to NSPS is 500 ppm NO_x (measured as NO_2). Performance Specification 2 is presented in Appendix B of this document.

As in performance testing, the objective is to obtain a representative and valid test. Portions of the monitor performance evaluation may be conducted concurrently with a performance test conducted under Section 60.8. A pretest protocol is highly recommended to familiarize the agency representative with the scheduling of the various tests. In addition to obtaining the production data noted previously, the agency representative may wish to observe various aspects of the test to observe the method in which each of the requirements is checked. A schematic of the monitor sampling system and the operation manual for the continuous monitor(s) may be useful to the agency representative.

SECTION 7

INSPECTION PROCEDURES

Field enforcement personnel must make periodic inspections of the nitric acid production facilities. It should be noted that a distinction is made between a followup inspection and observation of the performance tests although the data obtained from each are useful in determining the compliance status of the plant.

With the exception of performing visible emissions observations, a field inspector can determine very little from physically examining the mechanical components of the nitric acid process other than noting any major changes in equipment placement or fugitive emissions. Although records kept by the plant owner or operator will provide information to the field inspector so that an evaluation of the compliance status and the effectiveness of the maintenance may be conducted, the major emphasis of the inspection should be placed on reviewing continuous monitor outputs and operating records.

7.1 PLANT INSPECTION

7.1.1 Plant Entry

Because a major emphasis is placed on using available records to evaluate the performance of the nitric acid plant, the plant management must be contacted. The inspector should present his or her credentials and state the purpose of the inspection. In the interview with plant management the inspector should be able to determine whether the plant is operating, the production rate, and acid strength.

It should be noted that some of the information obtained during the inspections may be considered confidential by plant management. Ammonia feed rate, conversion efficiencies, and operating pressures are examples of data that plant managers often consider confidential because of economic and competitive reasons. These data should be handled in the manner as directed by Section 114(c) of the Clean Air Act, which specifies the rights and responsibilities of field inspectors in obtaining confidential data. These data, however, when compared with the same data obtained during performance testing or continuous monitor performance evaluation, will allow the field inspector to assess if any significant change in plant operation has occurred.

7.1.2 Inspection of the Plant

After ascertaining that the nitric acid plant is operating, the inspector should physically examine the plant. The inspector should start with an evaluation of any visible emissions that are present at the tail gas stack. The inspector should also check for any obvious changes in process equipment that may affect plant operation.

Proper operation of the continuous monitor is extremely important to the field inspector in determining the existence of excess emissions. The inspection should include a check of the following areas:

1. Is the monitor sited at the same point as during the monitor performance evaluation and any previous inspections?
2. Is the monitor the same (same model and serial number) as the one tested in the most recent performance evaluation?
3. What is the location of calibration gas injection or the method of required daily calibration checks?
4. What is the value of the span gas or cells used in daily calibration?

The continuous monitor display may be located near the analyzer or it may be placed in the control room or both. Typically the display will be in the control room with the other process instrumentation. A notation on the strip chart may be useful to the inspector if data from the day of the inspection is analyzed at a later date.

At this point, the inspector should obtain the readings from the nitric acid plant instrument panels. This would include, if possible, the level of NO_2 in the tail gas (ppm), the ammonia feed rate and/or the feed air volumetric flow rate, the operating temperatures and pressures, and the operating parameters monitored for any tail gas control device. Comparison of these values with the baseline values established during the performance test can aid in determining the compliance status. Methods for estimating emission rates are discussed in Section 7.2.

The inspector should also review operator logs and maintenance records to determine if any problems are recurring that have an unfavorable impact on the emissions. Daily production rates and operating hours should be scanned to determine if the owner or operator is meeting or exceeding any production limitations or restrictions listed in an operating permit. In addition, the field inspector should determine if the production rate is relatively constant and establish some value for average daily production. Although the conversion factors and figures presented in Section 7.2 are relatively accurate, the field inspector may wish to use the individual conversion rates established during the performance test for each nitric acid plant.

If the inspector establishes from the operating records that the nitric acid production rate is relatively constant from day to day and establishes an average value for production, the inspector may also establish a value in ppm NO_2 above which the NO_2 level exceeds the standard of 1.5 kg/metric ton. Establishing this level allows the inspector to quickly scan the data from the continuous monitor strip charts to determine if any potential excess emission period has occurred. If a set of values is discovered above this level, the production rate for those days should be used and not the average value as established above.

This procedure will quickly identify potential excess emission periods, although it will not identify all of these periods. This may only be accomplished through checking the daily records and comparing the averages of each 3-h period with the specified emission limit indicated in ppm.

The inspector will also need to verify that a daily zero and span calibration check is performed. This will usually be displayed as a square spike on the strip chart. The lack of daily zero and span calibration should be noted for the periods when the monitor is operating. If the monitor does not have an automatic zero and span calibration cycle, it is likely that the daily zero and span calibrations may be missed. If, however, the monitor is calibrated infrequently, then the data recorded on the monitor output may be in error. The options available to the field inspector are citing the owner or operator for not performing daily calibration checks and requiring the owner or operator to conduct a new performance evaluation of the monitor to determine if the monitor still fulfills the requirements of Performance Specification 2, Appendix B of the NSPS.

As with most pieces of equipment, continuous monitors can and do fail. The times when the monitor is inoperative are required to be recorded (Section 60.7(c)(3)) along with a notation with respect to the nature of the repairs and/or adjustments that were necessary to make the monitor operable again. The agency must decide if the monitor downtime is excessive and what actions should be taken to remedy any problems with monitor operation.

7.2 ESTIMATION OF EMISSIONS FROM NITRIC ACID PLANTS

An estimate of the tail gas volumetric flow rate within an accuracy of ±5 percent can be calculated from the air feed rate for an ammonia oxidation nitric acid plant. This is possible because the closed system design of the pressure process allows no gas loss or gain from the beginning introduction point to the exit exhaust stack. The oxygen content of the air is the only component in the air that is used in the reaction and it is carefully controlled to an excess of approximately 2 to 5 percent. Therefore, a definite ratio exists between the stack volumetric flow rate and the feed air volumetric flow rate. A small portion (4 to 10 percent) of the ammonia feed is reduced to inert nitrogen by the side reactions in the converter, but this amount is relatively constant and does not significantly affect the ratio of stack flow rate to feed air flow rate.

Furthermore, with the ammonia efficiency (sometimes expressed as nitrogen efficiency) being constant, the production rate of nitric acid (as 100 percent concentration) can also be estimated from the air feed rate. Because nitric acid plants are usually run at ammonia efficiencies of between 90 to 95 percent, an average of 93 can be used.

The graphs shown in Figures 28 and 29 were constructed based on the average data reported for numerous nitric acid plants under various operating conditions. These graphs can be used to estimate the stack volumetric flow rate and the plant emission rate in the following manner.

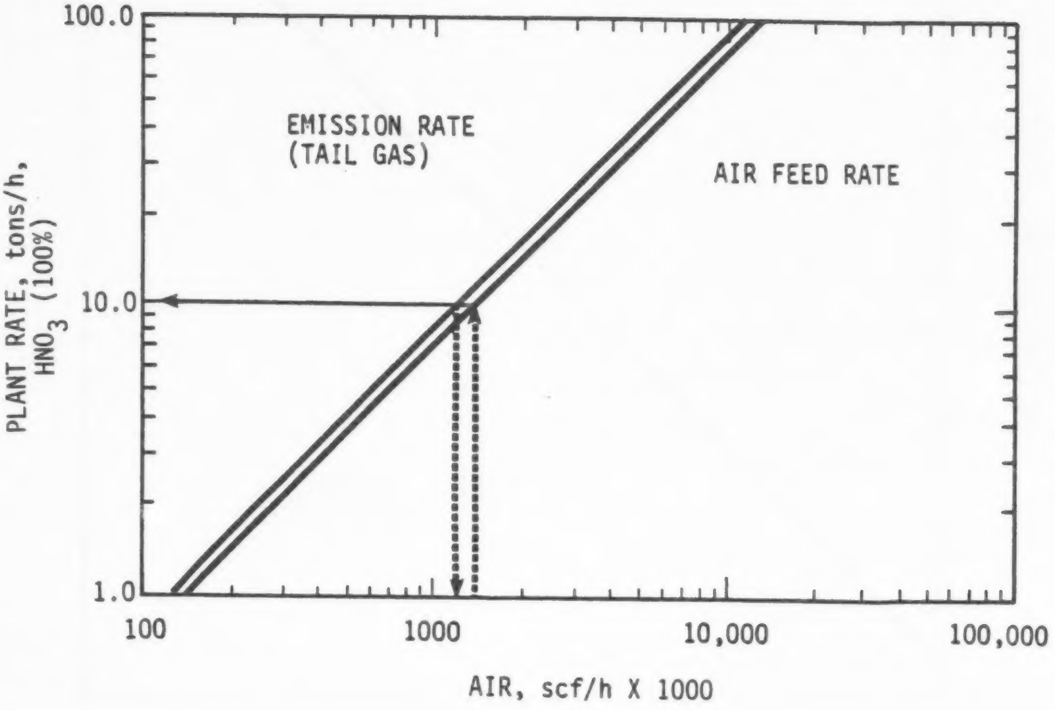


Figure 28. Production rate vs. air feed rate.

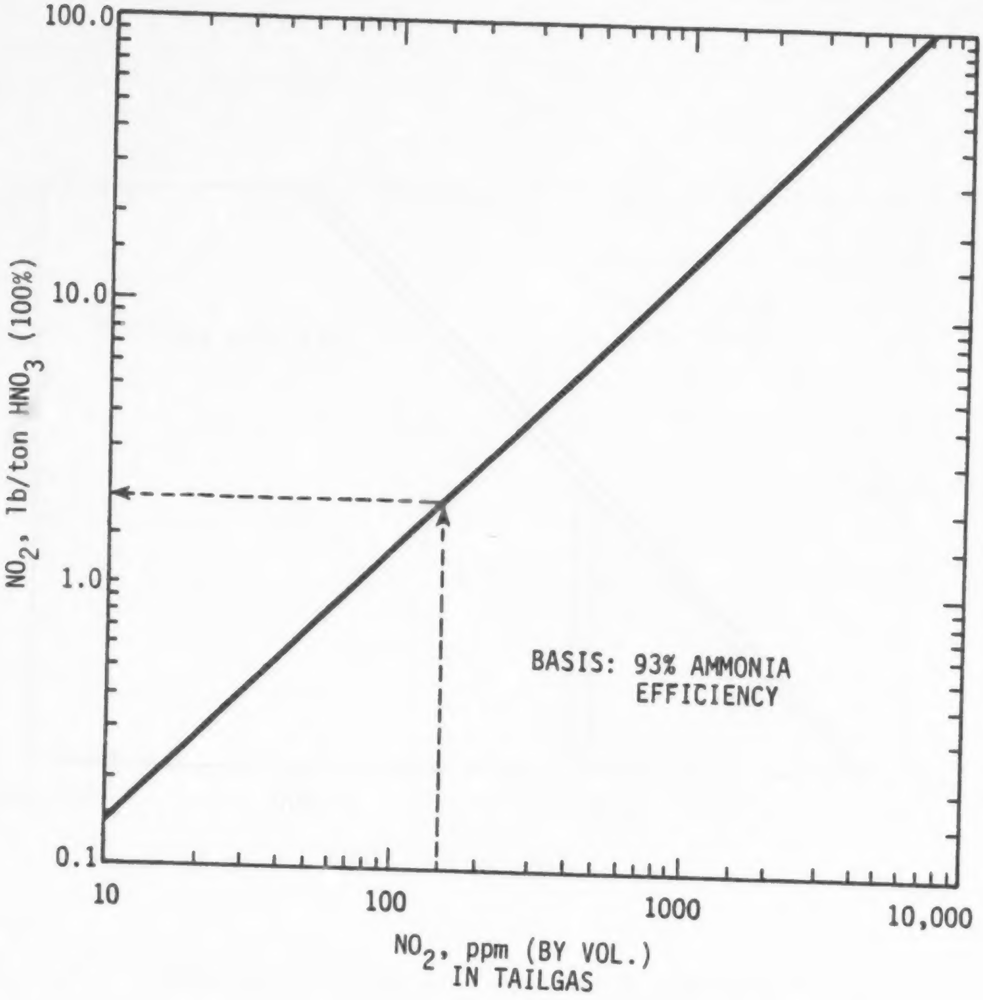


Figure 29. Conversion of NO_x tail gas concentration to mass emission.

1. Read the air flow rate to the converter (F1, usually in standard cubic foot per hour (scf/h).
2. Read the bleach air flow rate (F2, usually in scf/h).
3. Add the two air flows; this is the total air to the process. Assume this figure is 1,443,400 scf/h from Figure 28, the plant production rate is 10 tons/h HNO_3 (100 percent) and the stack volumetric flow rate is 1,209,000 scf/h.
4. Read the continuous nitrogen oxides monitor. Assume it reads 150 ppm NO_2; from Figure 29, the emission rate is 2.1 lb NO_2/ton HNO_3 (100 percent).

The graph shown in Figure 30 is useful to check ammonia efficiency and/or production rate when the plant ammonia feed rate is known. Before using the curves in Figure 30, one must know either the ammonia efficiency (nitrogen efficiency) or accurate ammonia and production rate values.

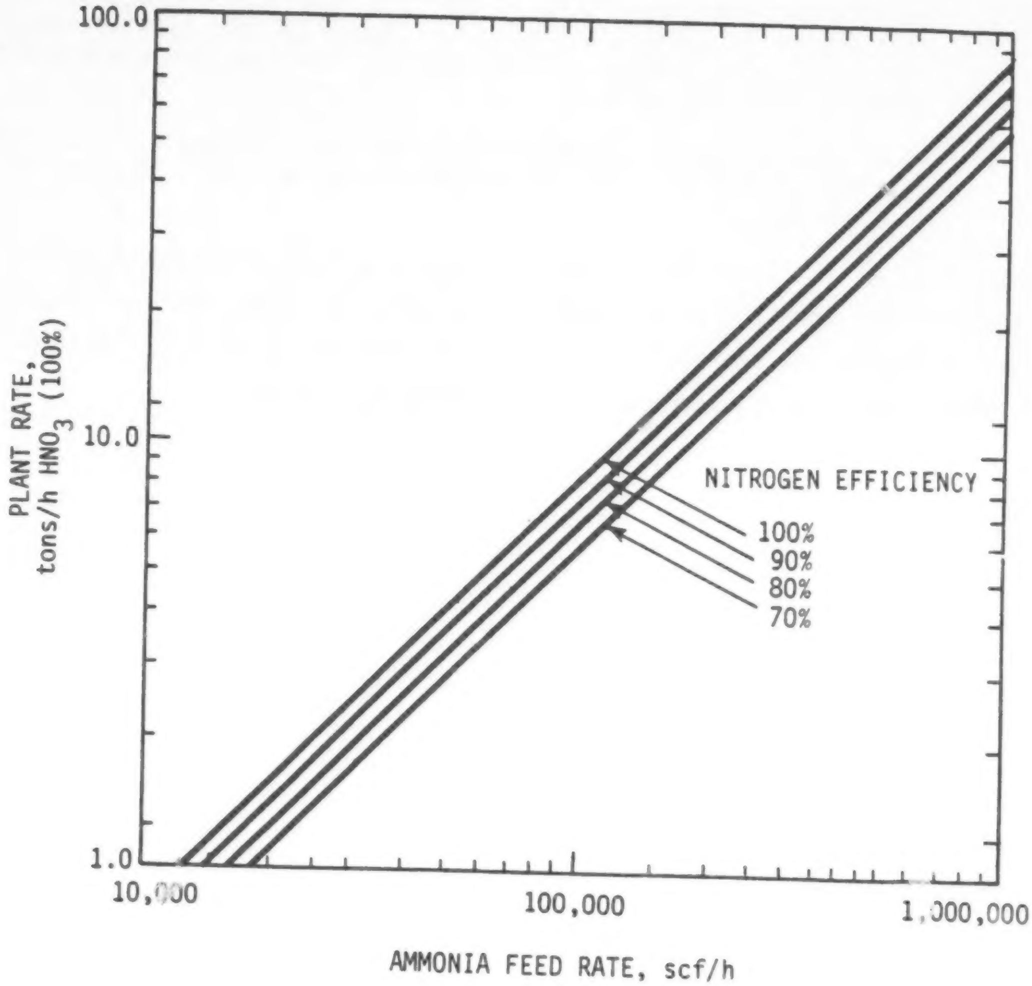


Figure 30. Ammonia feed rate vs. production rate.

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APPENDIX A

NSPS REGULATIONS

Subpart G—Standards of Performance for Nitric Acid Plants

§ 60.70 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to each nitric acid production unit, which is the affected facility.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

(Secs. 111 and 301(a) of the Clean Air Act; sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 2 of Pub. L. 90-148, 81 Stat. 504 (42 U.S.C. 1857c-6, 1857g(a)))

[42 FR 37936, July 25, 1977]

§ 60.71 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Nitric acid production unit" means any facility producing weak nitric acid by either the pressure or atmospheric pressure process.

(b) "Weak nitric acid" means acid which is 30 to 70 percent in strength.

§ 60.72 Standard for nitrogen oxides.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which:

(1) Contain nitrogen oxides, expressed as NO_2, in excess of 1.5 kg per metric ton of acid produced (3.0 lb per ton), the production being expressed as 100 percent nitric acid.

(2) Exhibit 10 percent opacity, or greater.

[39 FR 20794, June 14, 1974, as amended at 40 FR 46258, Oct. 6, 1975]

§ 60.73 Emission monitoring.

(a) A continuous monitoring system for the measurement of nitrogen oxides shall be installed, calibrated, maintained, and operated by the owner or operator. The pollutant gas used to prepare calibration gas mixtures under paragraph 2.1, Performance Specification 2 and for calibration checks under § 60.13(d) to this part, shall be nitrogen dioxide (NO_2). The span shall be set at 500 ppm of nitrogen dioxide. Reference Method 7 shall be used for conducting monitoring system performance evaluations under § 60.13(c).

(b) The owner or operator shall establish a conversion factor for the purpose of converting monitoring data into units of the applicable standard (kg/metric ton, lb/short ton). The conversion factor shall be established by measuring emissions with the continuous monitoring system concurrent with measuring emissions with the applicable reference method tests. Using only that portion of the continuous monitoring emission data that represents emission measurements concurrent with the reference method test periods, the conversion factor shall be determined by dividing the reference method test data averages by the monitoring data averages to obtain a ratio expressed in units of the applicable standard to units of the monitoring data, i.e., kg/metric ton per ppm (lb/short ton per ppm). The conversion factor shall be reestablished during any performance test under § 60.8 or any continuous monitoring system performance evaluation under § 60.13(c).

(c) The owner or operator shall record the daily production rate and hours of operation.

(d) [Reserved]

(e) For the purpose of reports required under § 60.7(c), periods of excess emissions that shall be reported are defined as any 3-hour period during which the average nitrogen oxides emissions (arithmetic average of three contiguous 1-hour periods) as measured by a continuous monitoring system exceed the standard under § 60.72(a).

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[39 FR 20794, June 14, 1974, as amended at 40 FR 46258, Oct. 6, 1975; 43 FR 8800, Mar. 3, 1978]

§ 60.74 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standard prescribed in § 60.72 as follows:

(1) Method 7 for the concentration of NO_x;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) For Method 7, the sample site shall be selected according to Method 1 and the sampling point shall be the centroid of the stack or duct or at a point no closer to the walls than 1 m (3.28 ft). Each run shall consist of at least four grab samples taken at approximately 15-minute intervals. The arithmetic mean of the samples shall constitute the run value. A velocity traverse shall be performed once per run.

(c) Acid production rate, expressed in metric tons per hour of 100 percent nitric acid, shall be determined during each testing period by suitable methods and shall be confirmed by a material balance over the production system.

(d) For each run, nitrogen oxides, expressed in g/metric ton of 100 percent nitric acid, shall be determined by dividing the emission rate in g/hr by the acid production rate. The emission rate shall be determined by the equation,

$$g/hr = Q_s \times c$$

where Q_s=volumetric flow rate of the effluent in dscm/hr, as determined in accordance with paragraph (a)(3) of this section, and $c = NO_x$ concentration in g/dscm, as determined in accordance with paragraph (a)(1) of this section.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[39 FR 20794, June 14, 1974, as amended at 43 FR 8800, Mar. 3, 1978]

Subpart H—Standards of Performance for Sulfuric Acid Plants

§ 60.80 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to each sulfuric acid production unit, which is the affected facility.

(b) Any facility under paragraph (a) of this section that commences construction or modification after August 17, 1971, is subject to the requirements of this subpart.

(Secs. 111 and 301(a) of the Clean Air Act; sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 2 of Pub. L. 90-148, 81 Stat. 504 (42 U.S.C. 1857c-6, 1857g(a)))

[42 FR 37936, July 25, 1977]

§ 60.81 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Sulfuric acid production unit" means any facility producing sulfuric acid by the contact process by burning elemental sulfur, alkylation acid, hydrogen sulfide, organic sulfides and mercaptans, or acid sludge, but does not include facilities where conversion to sulfuric acid is utilized primarily as a means of preventing emissions to the atmosphere of sulfur dioxide or other sulfur compounds.

(b) "Acid mist" means sulfuric acid mist, as measured by Method 8 of Appendix A to this part or an equivalent or alternative method.

[36 FR 24877, Dec. 23, 1971, as amended at 39 FR 20794, June 14, 1974]

§ 60.82 Standard for sulfur dioxide.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which contain sulfur dioxide in excess of 2 kg per metric ton of acid produced (4 lb per ton), the production being expressed as 100 percent H_2SO_4.

[39 FR 20794, June 14, 1974]

§ 60.83 Standard for acid mist.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall cause to be discharged into the atmosphere from any affected facility any gases which:

(1) Contain acid mist, expressed as H_2SO_4, in excess of 0.075 kg per metric ton of acid produced (0.15 lb per ton), the production being expressed as 100 percent H_2SO_4.

(2) Exhibit 10 percent opacity, or greater.

[39 FR 20794, June 14, 1974, as amended at 40 FR 46258, Oct. 6, 1975]

§ 60.84 Emission monitoring.

(a) A continuous monitoring system for the measurement of sulfur dioxide shall be installed, calibrated, maintained, and operated by the owner or operator. The pollutant gas used to prepare calibration gas mixtures under paragraph 2.1, Performance Specification 2 and for calibration checks under § 60.13(d), shall be sulfur dioxide (SO_2). Reference Method 8 shall be used for conducting monitoring system performance evaluations under § 60.13(c) except that only the sulfur dioxide portion of the Method 8 results shall be used. The span shall be set at 1000 ppm of sulfur dioxide.

(b) The owner or operator shall establish a conversion factor for the purpose of converting monitoring data into units of the applicable standard (kg/metric ton, lb/short ton). The conversion factor shall be determined, as a minimum, three times daily by measuring the concentration of sulfur dioxide entering the converter using suitable methods (e.g., the Reich test, National Air Pollution Control Administration Publication No. 999-AP-13) and calculating the appropriate conversion factor for each eight-hour period as follows:

$$CF = k[1.000 - 0.015r/r - s]$$

where:

CF = conversion factor (kg/metric ton per ppm, lb/short ton per ppm).

k = constant derived from material balance. For determining CF in metric units, $k = 0.0653$. For determining CF in English units, $k = 0.1306$.

r = percentage of sulfur dioxide by volume entering the gas converter. Appropriate corrections must be made for air injection plants subject to the Administrator's approval.

s = percentage of sulfur dioxide by volume in the emissions to the atmosphere determined by the continuous monitoring system required under paragraph (a) of this section.

(c) The owner or operator shall record all conversion factors and values under paragraph (b) of this section from which they were computed (i.e., CF, r, and s).

(d) [Reserved]

(e) For the purpose of reports under § 60.7(c), periods of excess emissions shall be all three-hour periods (or the arithmetic average of three consecutive one-hour periods) during which the integrated average sulfur dioxide emissions exceed the applicable standards under § 60.82.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[39 FR 20794, June 14, 1974, as amended at 40 FR 46258, Oct. 6, 1975; 43 FR 8800, Mar. 3, 1978]

§ 60.85 Test methods and procedures.

(a) The reference methods in Appendix A to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in §§ 60.82 and 60.83 as follows:

(1) Method 8 for the concentrations of SO_3 and acid mist;

(2) Method 1 for sample and velocity traverses;

(3) Method 2 for velocity and volumetric flow rate; and

(4) Method 3 for gas analysis.

(b) The moisture content can be considered to be zero. For Method 8 the sampling time for each run shall be at least 60 minutes and the minimum sample volume shall be 1.15 dscm (40.6 dscf) except that smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the Administrator.

(c) Acid production rate, expressed in metric tons per hour of 100 percent H_2SO_4, shall be determined during each testing period by suitable methods and shall be confirmed by a material balance over the production system.

(d) Acid mist and sulfur dioxide emissions, expressed in g/metric ton of 100 percent H_2SO_4, shall be determined by dividing the emission rate in g/hr by the acid production rate. The emission rate shall be determined by the equation, $g/hr = Q_s \times c$, where Q_s = volumetric flow rate of the effluent in dscm/hr as determined in accordance with paragraph (a)(3) of this section, and c = acid mist and SO_2 concentrations in g/dscm as determined in accordance with paragraph (a)(1) of this section.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[39 FR 20794, June 14, 1974, as amended at 43 FR 8800, Mar. 3, 1978]

Subpart I—Standards of Performance for Asphalt Concrete Plants

§ 60.90 Applicability and designation of affected facility.

(a) The affected facility to which the provisions of this subpart apply is each asphalt concrete plant. For the purpose of this subpart, an asphalt concrete plant is comprised only of any combination of the following: Dryers; systems for screening, handling, storing, and weighing hot aggregate; systems for loading, transferring, and storing mineral filler; systems for mixing asphalt concrete; and the loading, transfer, and storage systems associated with emission control systems.

(b) Any facility under paragraph (a) of this section that commences construction or modification after June 11, 1973, is subject to the requirements of this subpart.

(Secs. 111 and 301(a) of the Clean Air Act; sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 2 of Pub. L. 90-148, 81 Stat. 504 (42 U.S.C. 1857c-6, 1857g(a)))

[42 FR 37936, July 25, 1977]

§ 60.91 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A of this part.

(a) "Asphalt concrete plant" means any facility, as described in § 60.90, used to manufacture asphalt concrete by heating and drying aggregate and mixing with asphalt cements.

§ 60.92 Standard for particulate matter.

(a) On and after the date on which the performance test required to be conducted by § 60.8 is completed, no owner or operator subject to the provisions of this subpart shall discharge or cause the discharge into the atmosphere from any affected facility any gases which:

(1) Contain particulate matter in excess of 90 mg/dscm (0.04 gr/dscf).

(2) Exhibit 20 percent opacity, or greater.

[39 FR 9314, Mar. 8, 1974, as amended at 40 FR 46259, Oct. 6, 1975]

§ 60.93 Test methods and procedures.

(a) The reference methods appended to this part, except as provided for in § 60.8(b), shall be used to determine compliance with the standards prescribed in § 60.92 as follows:

(1) Method 5 for the concentration of particulate matter and the associated moisture content,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate, and

(4) Method 3 for gas analysis.

(b) For Method 5, the sampling time for each run shall be at least 60 minutes and the sampling rate shall be at least 0.9 dscm/hr (0.53 dscf/min) except that shorter sampling times, when necessitated by process variables or other factors, may be approved by the Administrator.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[39 FR 9314, Mar. 8, 1974, as amended at 43 FR 8800, Mar. 3, 1978]

Subpart J—Standards of Performance for Petroleum Refineries

§ 60.100 Applicability and designation of affected facility.

(a) The provisions of this subpart are applicable to the following affected facilities in petroleum refineries: fluid catalytic cracking unit catalyst regenerators, fuel gas combustion devices, and all Claus sulfur recovery plants except Claus plants of 20 long tons per day (LTD) or less. The Claus sulfur recovery plant need not be physically located within the boundaries of a petroleum refinery to be an affected facility, provided it processes gases produced within a petroleum refinery.

(b) Any fluid catalytic cracking unit catalyst regenerator or fuel gas combustion device under paragraph (a) of this section which commences construction or modification after June 11, 1973, or any Claus sulfur recovery plant under paragraph (a) of this section which commences construction or modification after October 4, 1976, is subject to the requirements of this part.

(Secs. 111, 114, 301(a), Clean Air Act, as amended (42 U.S.C. 7411, 7414, 7601(a)))

[43 FR 10868, Mar. 15, 1978, as amended at 4 FR 61543, Oct. 25, 1979]

§ 60.101 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act and in Subpart A.

(a) "Petroleum refinery" means any facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products

Subpart A—General Provisions

§ 60.1 Applicability.

Except as provided in Subparts B and C, the provisions of this part apply to the owner or operator of any stationary source which contains an affected facility, the construction or modification of which is commenced after the date of publication in this part of any standard (or, if earlier, the date of publication of any proposed standard) applicable to that facility.

[40 FR 53346, Nov. 17, 1975]

§ 60.2 Definitions.

The terms used in this part are defined in the Act or in this section as follows:

"Act" means the Clean Air Act (42 U.S.C. 1857 et seq., as amended by Pub. L. 91-604, 84 Stat. 1676).

"Administrator" means the Administrator of the Environmental Protection Agency or his authorized representative.

"Affected facility" means, with reference to a stationary source, any apparatus to which a standard is applicable.

"Alternative method" means any method of sampling and analyzing for an air pollutant which is not a reference or equivalent method but which has been demonstrated to the Administrator's satisfaction to, in specific cases, produce results adequate for his determination of compliance.

"Capital expenditure" means an expenditure for a physical or operational change to an existing facility which exceeds the product of the applicable "annual asset guideline repair allowance percentage" specified in the latest edition of Internal Revenue Service (IRS) Publication 534 and the existing facility's basis, as defined by section 1012 of the Internal Revenue Code. However, the total expenditure for a physical or operational change to an existing facility must not be reduced by any "excluded additions" as defined in IRS Publication 534, as would be done for tax purposes.

"Commenced" means, with respect to the definition of "new source" in section 111(a)(2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

"Construction" means fabrication, erection, or installation of an affected facility.

"Continuous monitoring system" means the total equipment, required under the emission monitoring sections in applicable subparts, used to sample and condition (if applicable), to analyze, and to provide a permanent record of emissions or process parameters.

"Equivalent method" means any method of sampling and analyzing for an air pollutant which has been demonstrated to the Administrator's satisfaction to have a consistent and quantitatively known relationship to the reference method, under specified conditions.

"Existing facility" means, with reference to a stationary source, any apparatus of the type for which a standard is promulgated in this part, and the construction or modification of which was commenced before the date of proposal of that standard; or any apparatus which could be altered in such a way as to be of that type.

"Isokinetic sampling" means sampling in which the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

"Malfunction" means any sudden and unavoidable failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner. Failures that are caused entirely or in part by poor maintenance, careless operation, or any other preventable upset condition or preventable equipment breakdown shall not be considered malfunctions.

"Modification" means any physical change in, or change in the method of operation of, an existing facility which increases the amount of any air pollutant (to which a standard applies) emitted into the atmosphere by that facility or which results in the emission of any air pollutant (to which a standard applies) into the atmosphere not previously emitted.

"Monitoring device" means the total equipment, required under the monitoring of operations sections in applicable subparts, used to measure and record (if applicable) process parameters.

"Nitrogen oxides" means all oxides of nitrogen except nitrous oxide, as measured by test methods set forth in this part.

"One-hour period" means any 60-minute period commencing on the hour.

"Opacity" means the degree to which emissions reduce the transmission of light and obscure the view of an object in the background.

"Owner or operator" means any person who owns, leases, operates, controls, or supervises an affected facility or a stationary source of which an affected facility is a part.

"Particulate matter" means any finely divided solid or liquid material, other than uncombined water, as measured by the reference methods specified under each applicable subpart, or an equivalent or alternative method.

"Proportional sampling" means sampling at a rate that produces a constant ration of sampling rate to stack gas flow rate.

"Reference method" means any method of sampling and analyzing for an air pollutant as described in Appendix A to this part.

"Run" means the net period of time during which an emission sample is collected. Unless otherwise specified, a run may be either intermittent or continuous within the limits of good engineering practice.

"Shutdown" means the cessation of operation of an affected facility for any purpose.

"Six-minute period" means any one of the 10 equal parts of a one-hour period.

"Standard" means a standard of performance proposed or promulgated under this part.

"Standard conditions" means a temperature of 293 K (68°F) and a pressure of 101.3 kilopascals (29.92 in Hg).

"Startup" means the setting in operation of an affected facility for any purpose.

"Volatile Organic Compound" means any organic compound which participates in atmospheric photochemical reactions; or which is measured by a reference method, an equivalent method, an alternative method, or which is determined by procedures specified under any subpart.

[44 FR 55173, Sept. 25, 1979, as amended at 45 FR 5617, Jan. 23, 1980; 45 FR 85415, Dec. 24, 1980]

§ 60.3 Units and abbreviations.

Used in this part are abbreviations and symbols of units of measure. These are defined as follows:

(a) System International (SI) units of measure:

A—ampere
g—gram
Hz—hertz
J—joule
K—degree Kelvin
kg—kilogram
m—meter
m^3—cubic meter
mg—milligram—10^{-3} gram
mm—millimeter—10^{-3} meter
Mg—megagram—10^{6} gram
mol—mole
N—newton
ng—nanogram—10^{-9} gram
nm—nanometer—10^{-9} meter
Pa—pascal
s—second
V—volt
W—watt
Ω—ohm
μg—microgram—10^{-6} gram

(b) Other units of measure:

Btu—British thermal unit
°C—degree Celsius (centigrade)
cal—calorie
cfm—cubic feet per minute
cu ft—cubic feet
dcf—dry cubic feet
dcm—dry cubic meter
dscf—dry cubic feet at standard conditions
dscm—dry cubic meter at standard conditions
eq—equivalent

°F—degree Fahrenheit
ft—feet
gal—gallon
gr—grain
g-eq—gram equivalent
hr—hour
in—inch
k—1,000
l—liter
lpm—liter per minute
lb—pound
meq—milliequivalent
min—minute
ml—milliliter
mol. wt.—molecular weight
ppb—parts per billion
ppm—parts per million
psia—pounds per square inch absolute
psig—pounds per square inch gage
°R—degree Rankine
scf—cubic feet at standard conditions
scfh—cubic feet per hour at standard conditions
scm—cubic meter at standard conditions
sec—second
sq ft—square feet
std—at standard conditions

(c) Chemical nomenclature:

CdS—cadmium sulfide
CO—carbon monoxide
CO_2—carbon dioxide
HCl—hydrochloric acid
Hg—mercury
H_2O—water
H_2S—hydrogen sulfide
H_2SO_4—sulfuric acid
N_2—nitrogen
NO—nitric oxide
NO_2—nitrogen dioxide
NO_x—nitrogen oxides
O_2—oxygen
SO_2—sulfur dioxide
SO_3—sulfur trioxide
SO_x—sulfur oxides

(d) Miscellaneous:

A.S.T.M.—American Society for Testing and Materials

(Secs. 111 and 301(a) of the Clean Air Act; sec. 4(a) of Pub. L. 91-604, 84 Stat. 1683; sec. 2 of Pub. L. 90-148, 81 Stat. 504 (42 U.S.C. 1857c-6, 1857g(a)))

[42 FR 37000, July 19, 1977; 42 FR 38178, July 27, 1977]

§ 60.4 Address.

(a) All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Enforcement Division. The regional offices are as follows:

Region I (Connecticut, Maine, New Hampshire, Massachusetts, Rhode Island, Vermont), John F. Kennedy Federal Building, Boston, Massachusetts 02203.

Region II (New York, New Jersey, Puerto Rico, Virgin Islands), Federal Office Building, 26 Federal Plaza (Foley Square), New York, New York 10007.

Region III (Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia), Curtis Building, Sixth and Walnut Streets, Philadelphia, Pennsylvania 19106.

Region IV (Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee), Suite 300, 1421 Peachtree Street, Atlanta, Georgia 30309.

Region V (Illinois, Indiana, Minnesota, Michigan, Ohio, Wisconsin), 230 South Dearborn Street, Chicago, Illinois 60604.

Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), 1600 Patterson Street, Dallas, Texas 75201.

Region VII (Iowa, Kansas, Missouri, Nebraska), 1735 Baltimore Street, Kansas City, Missouri 63108.

Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), 196 Lincoln Towers, 1860 Lincoln Street, Denver, Colorado 80203.

Region IX (Arizona, California, Hawaii, Nevada, Guam, American Samoa), 100 California Street, San Francisco, California 94111.

Region X (Washington, Oregon, Idaho, Alaska), 1200 Sixth Avenue, Seattle, Washington 98101.

(b) Section 111(c) directs the Administrator to delegate to each State, when appropriate, the authority to implement and enforce standards of performance for new stationary sources located in such State. All information required to be submitted to EPA under paragraph (a) of this section, must also be submitted to the appropriate State Agency of any State to which this authority has been delegated (provided, that each specific delegation may except sources from a certain Federal or State reporting requirement). The appropriate mailing address for those States whose delegation request has been approved is as follows:

(A) [Reserved]

(B) State of Alabama, Air Pollution Control Division, Air Pollution Control Commission, 645 S. McDonough Street, Montgomery, Alabama 36104.

(C) [Reserved]

(D) Arizona:

Maricopa County Department of Health Services, Bureau of Air Pollution Control, 1825 East Roosevelt Street, Phoenix, AZ 85006.

Pima County Health Department, Air Quality Control District, 151 West Congress, Tucson, AZ 85701.

Pima County Air Pollution Control District, 151 West Congress Street, Tucson, AZ 85701.

(E) [Reserved]

(F) California:

Bay Area Air Pollution Control District, 939 Ellis Street, San Francisco, CA 94109.
Del Norte County Air Pollution Control District, Courthouse, Crescent City, CA 95531.
Frensno County Air Pollution Control District, 515 S. Cedar Avenue, Fresno, CA 93702.
Humboldt County Air Pollution Control District, 5600 S. Broadway, Eureka, CA 95501.
Kern County Air Pollution Control District, 1700 Flower Street (P.O. Box 997), Bakersfield, CA 93302.
Madera County Air Pollution Control District, 135 W. Yosemite Avenue, Madera, CA 93637.
Mendocino County Air Pollution Control District, County Courthouse, Ukiah, CA 95482.
Monterey Bay Unified Air Pollution Control District, 420 Church Street (P.O. Box 487) Salinas, CA 93901.
Northern Sonoma County Air Pollution Control District, 3313 Chanate Road, Santa Rosa, CA 95404.
Sacramento County Air Pollution Control District, 3701 Branch Center Road, Sacramento, CA 95827.
San Diego County Air Pollution Control District, 9150 Chesapeake Drive, San Diego, CA 92123.
San Joaquin County Air Pollution Control District, 1601 E. Hazelton Street (P.O. Box 2009) Stockton, CA 95201.
Santa Barbara County Air Pollution Control District, 4440 Calle Real, Santa Barbara, CA 93110.
Shasta County Air Pollution Control District, 1855 Placer Street, Redding, CA 96001.
South Coast Air Quality Management District, 9420 Telstar Avenue, El Monte, CA 91731.
Stanislaus County Air Pollution Control District, 820 Scenic Drive, Modesto, CA 95350.
Trinity County Air Pollution Control District, Box AJ, Weaverville, CA 96093.
Ventura County Air Pollution Control District, 625 E. Santa Clara Street, Ventura, CA 93001.

(G) State of Colorado, Colorado Air Pollution Control Division, 4210 East 11th Avenue, Denver, Colorado 80220.

(H) State of Connecticut, Department of Environmental Protection, State Office Building, Hartford, Connecticut 06115.

(I) State of Delaware (for fossil fuel-fired steam generators; incinerators; nitric acid plants; asphalt concrete plants; storage vessels for petroleum liquids; sulfuric acid plants; sewage treatment plants; and electric utility steam generating units), Delaware Department of Natural Resources and Environmental Control, Edward Tatnall Building, Dover Delaware 19901.

(J)—(K) [Reserved]

(L) State of Georgia, Environmental Protection Division, Department of Natural Resources, 270 Washington Street, S.W., Atlanta, Georgia 30334.

(M) [Reserved]

(N) State of Idaho, Department of Health and Welfare, Statehouse, Boise, Idaho 83701.

(O) [Reserved]

(P) State of Indiana, Indiana Air Pollution Control Board, 1330 West Michigan Street, Indianapolis, Indiana 46206.

(Q) State of Iowa, Iowa Department of Environmental Quality, Henry A. Wallace Building, 900 East Grand, Des Moines, Iowa 50316.

(R) [Reserved]

(S) Division of Air Pollution Control, Department for Natural Resources and Environmental Protection, U.S. 127, Frankfort, Ky. 40601.

(T) [Reserved]

(U) State of Maine, Department of Environmental Protection, State House, Augusta, Maine 04330.

(V) State of Maryland: Bureau of Air Quality and Noise Control, Maryland State Department of Health and Mental Hygiene, 201 West Preston Street, Baltimore, Maryland 21201.

(W) Massachusetts Department of Environmental Quality Engineering, Division of Air Quality Control, 600 Washington Street, Boston, Massachusetts 02111.

(X) State of Michigan, Air Pollution Control Division, Michigan Department of Natural Resources, Stevens T. Mason Building, 8th Floor, Lansing, Michigan 48926.

(Y) Minnesota Pollution Control Agency, Division of Air Quality, 1935 West County Road B-2, Roseville, Minn. 55113.

(Z) [Reserved]

(AA) Missouri Department of Natural Resources, Post Office Box 1368, Jefferson City, Missouri 65101.

(BB) State of Montana, Department of Health and Environmental Services, Cogswell Building, Helena, Mont. 59601.

(CC) Nebraska Department of Environmental Control, P.O. Box 94653, State House Station, Lincoln, Nebraska 68509.

(DD) Nevada:

Nevada Department of Conservation and Natural Resources, Division of Environmental Protection, 201 South Fall Street, Carson City, NV 89710.

Clark County County District Health Department, Air Pollution Control Division, 625 Shadow Lane, Las Vegas, NV 89106.

Washoe County District Health Department, Division of Environmental Protection, 10 Kirman Avenue, Reno, NV 89502.

(EE) New Hampshire Air Pollution Control Agency, Department of Health and Welfare, State Laboratory Building, Hazen Drive, Concord, New Hampshire 03301.

(FF) State of New Jersey: New Jersey Department of Environmental Protection, John Fitch Plaza, P.O. Box 2807, Trenton, New Jersey 08625.

(GG) [Reserved]

(HH) New York: New York State Department of Environmental Conservation, 50 Wolf Road, New York 12233, attention: Division of Air Resources.

(II) North Carolina Environmental Management Commission, Department of Natural and Economic Resources, Division of Environmental Management, P.O. Box 27687, Raleigh, North Carolina 27611. Attention: Air Quality Section.

(JJ) State of North Dakota, State Department of Health, State Capitol, Bismarck, North Dakota 58501.

(KK) Ohio—

Medina, Summit and Portage Counties; Director, Air Pollution Control, 177 South Broadway, Akron, Ohio 44308.

Stark County; Director, Air Pollution Control Division, Canton City Health Department, City Hall, 218 Cleveland Avenue SW., Canton, Ohio 44702.

Butler, Clermont, Hamilton and Warren Counties; Superintendent, Division of Air Pollution Control, 2400 Beekman Street, Cincinnati, Ohio 45214.

Cuyahoga County; Commissioner, Division of Air Pollution Control, Department of Public Health and Welfare, 2735 Broadway Avenue, Cleveland, Ohio 44115.

Lorain County; Control Officer, Division of Air Pollution Control, 200 West Erie Avenue, 7th Floor, Lorain, Ohio 44052.

Belmont, Carroll, Columbiana, Harrison, Jefferson, and Monroe Counties; Director, North Ohio Valley Air Authority (NOVAA), 814 Adams Street, Steubenville, Ohio 43952.

Clark, Darke, Greene, Miami, Montgomery, and Preble Counties; Supervisor, Regional Air Pollution Control Agency (RAPCA), Montgomery County Health Department, 451 West Third Street, Dayton, Ohio 45402.

Lucas County and the City of Rossford (in Wood County); Director, Toledo Pollution Control Agency, 26 Main Street, Toledo, Ohio 43605.

Adams, Brown, Lawrence, and Scioto Counties; Engineer-Director, Air Division, Portsmouth City Health Department, 740 Second Street, Portsmouth, Ohio 45662.

Allen, Ashland, Auglaize, Crawford, Defiance, Erie, Fulton, Hancock, Hardin, Henry, Huron, Knox, Marion, Mercer, Morrow, Ottawa, Paulding, Putnam, Richland, Sandusky, Seneca, Van Wert, Williams, Wood (except City of Rossford), and Wyandot Counties; Ohio Environmental Protection Agency, Northwest District Office, 111 West Washington Street, Bowling Green, Ohio 43402.

Ashtabula, Geauga, Lake, Mahoning, Trumbull, and Wayne Counties; Ohio Environmental Protection Agency, Northeast District Office, 2110 East Aurora Road, Twinsburg, Ohio 44087.

Athens, Coshocton, Gallia, Guernsey, Highland, Hocking, Holmes, Jackson, Meigs, Morgan, Muskingum, Noble, Perry, Pike, Ross, Tuscarawas, Vinton, and Washington Counties; Ohio Environmental Protection Agency, Southeast District Office, Route 3, Box 603, Logan, Ohio 43138.

Champaign, Clinton, Logan, and Shelby Counties; Ohio Environmental Protection Agency, Southwest District Office, 7 East Fourth Street, Dayton, Ohio 45402.

Delaware, Fairfield, Fayette, Franklin, Licking, Madison, Pickaway, and Union Counties; Ohio Environmental Protection Agency, Central District Office, 369 East Broad Street, Columbus, Ohio 43215.

(LL) [Reserved]

(MM) State of Oregon, Department of Environmental Quality, 1234 SW. Morrison Street, Portland, Oregon 97205.

(NN) (a) City of Philadelphia: Philadelphia Department of Public Health, Air Management Services, 801 Arch Street, Philadelphia, Pennsylvania 19107.

(b) Commonwealth of Pennsylvania, Department of Environmental Resources, Post Office Box 2063, Harrisburg, Pennsylvania 17120.

(OO) State of Rhode Island, Department of Environmental Management, 83 Park Street, Providence, Rhode Island 02908.

(PP) State of South Carolina, Office of Environmental Quality Control, Department of Health and Environmental Control, 2600 Bull Street, Columbia, South Carolina 29201.

(QQ) State of South Dakota, Department of Environmental Protection, Joe Foss Building, Pierre, South Dakota 57501.

(RR) Division of Air Pollution Control, Tennessee Department of Public Health, 256 Capitol Hill Building, Nashville, Tennessee 37219.

(SS) State of Texas, Texas Air Control Board, 8520 Shoal Creek Boulevard, Austin, Texas 78758

(TT) State of Utah, Utah Air Conservation Committee, State Division of Health, 44 Medical Drive, Salt Lake City, Utah 84113.

(UU) State of Vermont, Agency of Environmental Protection, Box 489, Montpelier, Vermont 05602.

(VV) Commonwealth of Virginia, Virginia State Air Pollution Control Board, Room 1106, Ninth Street Office Building, Richmond, Virginia 23219.

(WW) (i) Washington: State of Washington, Department of Ecology, Olympia, Washington 98504.

(ii) Northwest Air Pollution Authority, 207 Pioneer Building, Second and Pine Streets, Mount Vernon, Washington 98273.

(iii) Puget Sound Air Pollution Control Agency, 410 West Harrison Street, Seattle, Washington 98119.

(iv) Spokane County Air Pollution Control Authority, North 811 Jefferson, Spokane, Washington 99201.

(v) Southwest Air Pollution Control Authority, Suite 7601 H, NE Hazel Dell Avenue, Vancouver, Washington 98665.

(vi) Olympic Air Pollution Control Authority, 120 East State Avenue, Olympia, WA 98501.

(XX) [Reserved]

(YY) Wisconsin—Wisconsin Department of Natural Resources, P.O. Box 7921, Madison, Wisconsin 53707.

(ZZ) State of Wyoming, Air Quality Division of the Department of Environmental Quality, Hathaway Building, Cheyenne, Wyo. 82002.

(AAA) [Reserved]

(BBB) Commonwealth of Puerto Rico: Commonwealth of Puerto Rico Environmental Quality Board, P.O. Box 11785, Santurce, P.R. 00910.

(CCC) U.S. Virgin Islands: U.S. Virgin Islands Department of Conservation and Cultural Affairs, P.O. Box 578, Charlotte Amalie, St. Thomas, U.S. Virgin Islands 00801.

(Secs. 101, 110, 111, 112, 114, 160-169, 301, Clean Air Act as amended (42 U.S.C. 7401, 7410, 7411, 7412, 7414, 7470-7479, 7491, 7601))

[40 FR 18169, Apr. 25, 1975, as amended at 40 FR 26677, June 25, 1975]

NOTE: For amendments to § 60.4 see the List of CFR Sections Affected appearing in the Finding Aids section of this volume.

§ 60.5 Determination of construction or modification.

(a) When requested to do so by an owner or operator, the Administrator will make a determination of whether action taken or intended to be taken by such owner or operator constitutes construction (including reconstruction) or modification or the commencement thereof within the meaning of this part.

(b) The Administrator will respond to any request for a determination under paragraph (a) of this section within 30 days of receipt of such request.

[40 FR 58418, Dec. 16, 1975]

§ 60.6 Review of plans.

(a) When requested to do so by an owner or operator, the Administrator will review plans for construction or modification for the purpose of providing technical advice to the owner or operator.

(b)(1) A separate request shall be submitted for each construction or modification project.

(2) Each request shall identify the location of such project, and be accompanied by technical information describing the proposed nature, size, design, and method of operation of each affected facility involved in such project, including information on any equipment to be used for measurement or control of emissions.

(c) Neither a request for plans review nor advice furnished by the Administrator in response to such request shall (1) relieve an owner or operator of legal responsibility for compliance with any provision of this part or of any applicable State or local requirement, or (2) prevent the Administrator from implementing or enforcing any provision of this part or taking any other action authorized by the Act.

[36 FR 24877, Dec. 23, 1971, as amended at 39 FR 9314, Mar. 8, 1974]

§ 60.7 Notification and record keeping.

(a) Any owner or operator subject to the provisions of this part shall furnish the Administrator written notification as follows:

(1) A notification of the date construction (or reconstruction as defined under § 60.15) of an affected facility is commenced postmarked no later than 30 days after such date. This requirement shall not apply in the case of mass-produced facilities which are purchased in completed form.

(2) A notification of the anticipated date of initial startup of an affected facility postmarked not more than 60 days nor less than 30 days prior to such date.

(3) A notification of the actual date of initial startup of an affected facility postmarked within 15 days after such date.

(4) A notification of any physical or operational change to an existing facility which may increase the emission rate of any air pollutant to which a standard applies, unless that change is specifically exempted under an applicable subpart or in § 60.14(e). This notice shall be postmarked 60 days or as soon as practicable before the change is commenced and shall include information describing the precise nature of the change, present and proposed emission control systems, productive capacity of the facility before and after the change, and the expected completion date of the change. The Administrator may request additional relevant information subsequent to this notice.

(5) A notification of the date upon which demonstration of the continuous monitoring system performance commences in accordance with § 60.13(c). Notification shall be postmarked not less than 30 days prior to such date.

(b) Any owner or operator subject to the provisions of this part shall maintain records of the occurrence and duration of any startup, shutdown, or malfunction in the operation of an affected facility; any malfunction of the air pollution control equipment; or any periods during which a continuous monitoring system or monitoring device is inoperative.

(c) Each owner or operator required to install a continuous monitoring system shall submit a written report of excess emissions (as defined in applicable subparts) to the Administrator for every calendar quarter. All quarterly reports shall be postmarked by the 30th day following the end of each calendar quarter and shall include the following information:

(1) The magnitude of excess emissions computed in accordance with § 60.13(h), any conversion factor(s) used, and the date and time of commencement and completion of each time period of excess emissions.

(2) Specific identification of each period of excess emissions that occurs during startups, shutdowns, and malfunctions of the affected facility. The nature and cause of any malfunction (if known), the corrective action taken or preventative measures adopted.

(3) The date and time identifying each period during which the continuous monitoring system was inoperative except for zero and span checks and the nature of the system repairs or adjustments.

(4) When no excess emissions have occurred or the continuous monitoring system(s) have not been inoperative, repaired, or adjusted, such information shall be stated in the report.

(d) Any owner or operator subject to the provisions of this part shall maintain a file of all measurements, including continuous monitoring system, monitoring device, and performance testing measurements; all continuous monitoring system performance evaluations; all continuous monitoring system or monitoring device calibration checks; adjustments and maintenance performed on these systems or devices; and all other information required by this part recorded in a permanent form suitable for inspection. The file shall be retained for at least two years following the date of such measurements, maintenance, reports, and records.

(e) If notification substantially similar to that in paragraph (a) of this section is required by any other State or local agency, sending the Administrator a copy of that notification will satisfy the requirements of paragraph (a) of this section.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[36 FR 24877, Dec. 28, 1971, as amended at 40 FR 46254, Oct. 6, 1975; 40 FR 58418, Dec.

16, 1975; 43 FR 8800, Mar. 3, 1978; 45 FR 5617, Jan. 23, 1980]

§ 60.8 Performance tests.

(a) Within 60 days after achieving the maximum production rate at which the affected facility will be operated, but not later than 180 days after initial startup of such facility and at such other times as may be required by the Administrator under section 114 of the Act, the owner or operator of such facility shall conduct performance test(s) and furnish the Administrator a written report of the results of such performance test(s).

(b) Performance tests shall be conducted and data reduced in accordance with the test methods and procedures contained in each applicable subpart unless the Administrator (1) specifies or approves, in specific cases, the use of a reference method with minor changes in methodology, (2) approves the use of an equivalent method, (3) approves the use of an alternative method the results of which he has determined to be adequate for indicating whether a specific source is in compliance, or (4) waives the requirement for performance tests because the owner or operator of a source has demonstrated by other means to the Administrator's satisfaction that the affected facility is in compliance with the standard. Nothing in this paragraph shall be construed to abrogate the Administrator's authority to require testing under section 114 of the Act.

(c) Performance tests shall be conducted under such conditions as the Administrator shall specify to the plant operator based on representative performance of the affected facility. The owner or operator shall make available to the Administrator such records as may be necessary to determine the conditions of the performance tests. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a performance test nor shall emissions in excess of the level of the applicable emission limit during periods of startup, shutdown, and malfunction be considered a violation of the applicable emission limit unless otherwise specified in the applicable standard.

(d) The owner or operator of an affected facility shall provide the Administrator at least 30 days prior notice of any performance test, except as specified under other subparts, to afford the Administrator the opportunity to have an observer present.

(e) The owner or operator of an affected facility shall provide, or cause to be provided, performance testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to such facility.

(2) Safe sampling platform(s).

(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(f) Unless otherwise specified in the applicable subpart, each performance test shall consist of three separate runs using the applicable test method. Each run shall be conducted for the time and under the conditions specified in the applicable standard. For the purpose of determining compliance with an applicable standard, the arithmetic means of results of the three runs shall apply. In the event that a sample is accidentally lost or conditions occur in which one of the three runs must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sample train, extreme meteorological conditions, or other circumstances, beyond the owner or operator's control, compliance may, upon the Administrator's approval, be determined using the arithmetic mean of the results of the two other runs.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[36 FR 24877, Dec. 23, 1971, as amended at 39 FR 9314, Mar. 8, 1974; 42 FR 57126, Nov. 1, 1977; 43 FR 8800, Mar. 3, 1978; 44 FR 33612, June 11, 1979]

§ 60.9 Availability of information.

The availability to the public of information provided to, or otherwise obtained by, the Administrator under this Part shall be governed by Part 2 of this chapter. (Information submitted voluntarily to the Administrator for the purposes of §§ 60.5 and 60.6 is governed by § 2.201 through § 2.213 of this chapter and not by § 2.301 of this chapter.)

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[41 FR 36918, Sept. 1, 1976, as amended at 43 FR 8800, Mar. 3, 1978]

§ 60.10 State authority.

The provisions of this part shall not be construed in any manner to preclude any State or political subdivision thereof from:

(a) Adopting and enforcing any emission standard or limitation applicable to an affected facility, provided that such emission standard or limitation is not less stringent than the standard applicable to such facility.

(b) Requiring the owner or operator of an affected facility to obtain permits, licenses, or approvals prior to initiating construction, modification, or operation of such facility.

(Sec. 116, Clean Air Act as amended (42 U.S.C. 7416))

[36 FR 24877, Dec. 23, 1971, as amended at 43 FR 8800, Mar. 3, 1978]

§ 60.11 Compliance with standards and maintenance requirements.

(a) Compliance with standards in this part, other than opacity standards, shall be determined only by performance tests established by § 60.8, unless otherwise specified in the applicable standard.

(b) Compliance with opacity standards in this part shall be determined by conducting observations in accordance with Reference Method 9 in Appendix A of this part or any alternative method that is approved by the Administrator. Opacity readings of portions of plumes which contain condensed, uncombined water vapor shall not be used for purposes of determining compliance with opacity standards. The results of continuous monitoring by transmissometer which indicate that the opacity at the time visual observations were made was not in excess of the standard are probative but not conclusive evidence of the actual opacity of an emission, provided that the source shall meet the burden of proving that the instrument used meets (at the time of the alleged violation) Performance Specification 1 in Appendix B of this part, has been properly maintained and (at the time of the alleged violation) calibrated, and that the resulting data have not been tampered with in any way.

(c) The opacity standards set forth in this part shall apply at all times except during periods of startup, shutdown, malfunction, and as otherwise provided in the applicable standard.

(d) At all times, including periods of startup, shutdown, and malfunction, owners and operators shall, to the extent practicable, maintain and operate any affected facility including associated air pollution control equipment in a manner consistent with good air pollution control practice for minimizing emissions. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator which may include, but is not limited to, monitoring results, opacity observations, review of operating and maintenance procedures, and inspection of the source.

(e)(1) An owner or operator of an affected facility may request the Administrator to determine opacity of emissions from the affected facility during the initial performance tests required by § 60.8.

(2) Upon receipt from such owner or operator of the written report of the results of the performance tests required by § 60.8, the Administrator will make a finding concerning compliance with opacity and other applicable standards. If the Administrator finds that an affected facility is in compliance with all applicable standards for which performance tests are conducted in accordance with § 60.8 of this part but during the time such performance tests are being conducted fails to meet any applicable opacity standard, he shall notify the owner or operator and advise him that he may petition the Administrator within 10 days of receipt of notification to make appropriate adjustment to the opacity standard for the affected facility.

(3) The Administrator will grant such a petition upon a demonstration by the owner or operator that the affected facility and associated air pollu-

tion control equipment was operated and maintained in a manner to minimize the opacity of emissions during the performance tests; that the performance tests were performed under the conditions established by the Administrator; and that the affected facility and associated air pollution control equipment were incapable of being adjusted or operated to meet the applicable opacity standard.

(4) The Administrator will establish an opacity standard for the affected facility meeting the above requirements at a level at which the source will be able, as indicated by the performance and opacity tests, to meet the opacity standard at all times during which the source is meeting the mass or concentration emission standard. The Administrator will promulgate the new opacity standard in the FEDERAL REGISTER.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[38 FR 28565, Oct. 15, 1973, as amended at 39 FR 39873, Nov. 12, 1974; 42 FR 26206, May 23, 1977; 43 FR 8800, Mar. 3, 1978; 45 FR 23379, Apr. 4, 1980]

§ 60.12 Circumvention.

No owner or operator subject to the provisions of this part shall build, erect, install, or use any article, machine, equipment or process, the use of which conceals an emission which would otherwise constitute a violation of an applicable standard. Such concealment includes, but is not limited to, the use of gaseous diluents to achieve compliance with an opacity standard or with a standard which is based on the concentration of a pollutant in the gases discharged to the atmosphere.

[39 FR 9314, Mar. 8, 1974]

§ 60.13 Monitoring requirements.

(a) For the purposes of this section, all continuous monitoring systems required under applicable subparts shall be subject to the provisions of this section upon promulgation of performance specifications for continuous monitoring system under Appendix B to this part, unless:

(1) The continuous monitoring system is subject to the provisions of paragraphs (c)(2) and (c)(3) of this section, or

(2) otherwise specified in an applicable subpart or by the Administrator.

(b) All continuous monitoring systems and monitoring devices shall be installed and operational prior to conducting performance tests under § 60.8. Verification of operational status shall, as a minimum, consist of the following:

(1) For continuous monitoring systems referenced in paragraph (c)(1) of this section, completion of the conditioning period specified by applicable requirements in Appendix B.

(2) For continuous monitoring systems referenced in paragraph (c)(2) of this section, completion of seven days of operation.

(3) For monitoring devices referenced in applicable subparts, completion of the manufacturer's written requirements or recommendations for checking the operation or calibration of the device.

(c) During any performance tests required under § 60.8 or within 30 days thereafter and at such other times as may be required by the Administrator under section 114 of the Act, the owner or operator of any affected facility shall conduct continuous monitoring system performance evaluations and furnish the Administrator within 60 days thereof two or, upon request, more copies of a written report of the results of such tests. These continuous monitoring system performance evaluations shall be conducted in accordance with the following specifications and procedures:

(1) Continuous monitoring systems listed within this paragraph except as provided in paragraph (c)(2) of this section shall be evaluated in accordance with the requirements and procedures contained in the applicable performance specification of Appendix B as follows:

(i) Continuous monitoring systems for measuring opacity of emissions shall comply with Performance Specification 1.

(ii) Continuous monitoring systems for measuring nitrogen oxides emissions shall comply with Performance Specification 2.

(iii) Continuous monitoring systems for measuring sulfur dioxide emissions shall comply with Performance Specification 2.

(iv) Continuous monitoring systems for measuring the oxygen content or carbon dioxide content of effluent gases shall comply with Performance Specification 3.

(2) An owner or operator who, prior to September 11, 1974, entered into a binding contractual obligation to purchase specific continuous monitoring system components except as referenced by paragraph (c)(2)(iii) of this section shall comply with the following requirements:

(i) Continuous monitoring systems for measuring opacity of emissions shall be capable of measuring emission levels within ±20 percent with a confidence level of 95 percent. The Calibration Error Test and associated calculation procedures set forth in Performance Specification 1 of Appendix B shall be used for demonstrating compliance with this specification.

(ii) Continuous monitoring systems for measurement of nitrogen oxides or sulfur dioxide shall be capable of measuring emission levels within ±20 percent with a confidence level of 95 percent. The Calibration Error Test, the Field Test for Accuracy (Relative), and associated operating and calculation procedures set forth in Performance Specification 2 of Appendix B shall be used for demonstrating compliance with this specification.

(iii) Owners or operators of all continuous monitoring systems installed on an affected facility prior to October 6, 1975 are not required to conduct tests under paragraphs (c)(2)(i) and/or (ii) of this section unless requested by the Administrator.

(3) All continuous monitoring systems referenced by paragraph (c)(2) of this section shall be upgraded or replaced (if necessary) with new continuous monitoring systems, and the new or improved systems shall be demonstrated to comply with applicable performance specifications under paragraph (c)(1) of this section on or before September 11, 1979.

(d) Owners or operators of all continuous monitoring systems installed in accordance with the provisions of this part shall check the zero and span drift at least once daily in accordance with the method prescribed by the manufacturer of such systems unless the manufacturer recommends adjustments at shorter intervals, in which case such recommendations shall be followed. The zero and span shall, as a minimum, be adjusted whenever the 24-hour zero drift or 24-hour calibration drift limits of the applicable performance specifications in Appendix B are exceeded. For continuous monitoring systems measuring opacity of emissions, the optical surfaces exposed to the effluent gases shall be cleaned prior to performing the zero or span drift adjustments except that for systems using automatic zero adjustments, the optical surfaces shall be cleaned when the cumulative automatic zero compensation exceeds four percent opacity. Unless otherwise approved by the Administrator, the following procedures, as applicable, shall be followed:

(1) For extractive continuous monitoring systems measuring gases, minimum procedures shall include introducing applicable zero and span gas mixtures into the measurement system as near the probe as is practical. Span and zero gases certified by their manufacturer to be traceable to National Bureau of Standards reference gases shall be used whenever these reference gases are available. The span and zero gas mixtures shall be the same composition as specified in Appendix B of this part. Every six months from date of manufacture, span and zero gases shall be reanalyzed by conducting triplicate analyses with Reference Methods 6 for SO_2, 7 for NO_x, and 3 for O_2 and CO_2, respectively. The gases may be analyzed at less frequent intervals if longer shelf lives are guaranteed by the manufacturer.

(2) For non-extractive continuous monitoring systems measuring gases, minimum procedures shall include upscale check(s) using a certified calibration gas cell or test cell which is functionally equivalent to a known gas concentration. The zero check may be performed by computing the zero value from upscale measurements or

by mechanically producing a zero condition.

(3) For continuous monitoring systems measuring opacity of emissions, minimum procedures shall include a method for producing a simulated zero opacity condition and an upscale (span) opacity condition using a certified neutral density filter or other related technique to produce a known obscuration of the light beam. Such procedures shall provide a system check of the analyzer internal optical surfaces and all electronic circuitry including the lamp and photodetector assembly.

(e) Except for system breakdowns, repairs, calibration checks, and zero and span adjustments required under paragraph (d) of this section, all continuous monitoring systems shall be in continuous operation and shall meet minimum frequency of operation requirements as follows:

(1) All continuous monitoring systems referenced by paragraphs (c)(1) and (c)(2) of this section for measuring opacity of emissions shall complete a minimum of one cycle of sampling and analyzing for each successive ten-second period and one cycle of data recording for each successive six-minute period.

(2) All continuous monitoring systems referenced by paragraph (c)(1) of this section for measuring oxides of nitrogen, sulfur dioxide, carbon dioxide, or oxygen shall complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each successive 15-minute period.

(3) All continuous monitoring systems referenced by paragraph (c)(2) of this section, except opacity, shall complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each successive one-hour period.

(f) All continuous monitoring systems or monitoring devices shall be installed such that representative measurements of emissions or process parameters from the affected facility are obtained. Additional procedures for location of continuous monitoring systems contained in the applicable Performance Specifications of Appendix B of this part shall be used.

(g) When the effluents from a single affected facility or two or more affected facilities subject to the same emission standards are combined before being released to the atmosphere, the owner or operator may install applicable continuous monitoring systems on each effluent or on the combined effluent. When the affected facilities are not subject to the same emission standards, separate continuous monitoring systems shall be installed on each effluent. When the effluent from one affected facility is released to the atmosphere through more than one point, the owner or operator shall install applicable continuous monitoring systems on each separate effluent unless the installation of fewer systems is approved by the Administrator.

(h) Owners or operators of all continuous monitoring systems for measurement of opacity shall reduce all data to six-minute averages and for systems other than opacity to one-hour averages for time periods under § 60.2 (x) and (r) respectively. Six-minute opacity averages shall be calculated from 24 or more data points equally spaced over each six-minute period. For systems other than opacity, one-hour averages shall be computed from four or more data points equally spaced over each one-hour period. Data recorded during periods of system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in the data averages computed under this paragraph. An arithmetic or integrated average of all data may be used. The data output of all continuous monitoring systems may be recorded in reduced or nonreduced form (e.g. ppm pollutant and percent O_2 or lb/million Btu of pollutant). All excess emissions shall be converted into units of the standard using the applicable conversion procedures specified in subparts. After conversion into units of the standard, the data may be rounded to the same number of significant digits used in subparts to specify the applicable standard (e.g., rounded to the nearest one percent opacity).

(i) After receipt and consideration of written application, the Administrator may approve alternatives to any monitoring procedures or requirements of this part including, but not limited to the following:

(1) Alternative monitoring requirements when installation of a continuous monitoring system or monitoring device specified by this part would not provide accurate measurements due to liquid water or other interferences caused by substances with the effluent gases.

(2) Alternative monitoring requirements when the affected facility is infrequently operated.

(3) Alternative monitoring requirements to accommodate continuous monitoring systems that require additional measurements to correct for stack moisture conditions.

(4) Alternative locations for installing continuous monitoring systems or monitoring devices when the owner or operator can demonstrate that installation at alternate locations will enable accurate and representative measurements.

(5) Alternative methods of converting pollutant concentration measurements to units of the standards.

(6) Alternative procedures for performing daily checks of zero and span drift that do not involve use of span gases or test cells.

(7) Alternatives to the A.S.T.M. test methods or sampling procedures specified by any subpart.

(8) Alternative continuous monitoring systems that do not meet the design or performance requirements in Performance Specification 1, Appendix B, but adequately demonstrate a definite and consistent relationship between its measurements and the measurements of opacity by a system complying with the requirements in Performance Specification 1. The Administrator may require that such demonstration be performed for each affected facility.

(9) Alternative monitoring requirements when the effluent from a single affected facility or the combined effluent from two or more affected facilities are released to the atmosphere through more than one point.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

[40 FR 46255, Oct. 6, 1975; 40 FR 59205, Dec. 22, 1975, as amended at 41 FR 35185, Aug. 20, 1976; 42 FR 5936, Jan. 31, 1977; 43 FR 7572, Feb. 23, 1978; 43 FR 8800, Mar. 3, 1978]

§ 60.14 Modification.

(a) Except as provided under paragraphs (e) and (f) of this section, any physical or operational change to an existing facility which results in an increase in the emission rate to the atmosphere of any pollutant to which a standard applies shall be considered a modification within the meaning of section 111 of the Act. Upon modification, an existing facility shall become an affected facility for each pollutant to which a standard applies and for which there is an increase in the emission rate to the atmosphere.

(b) Emission rate shall be expressed as kg/hr of any pollutant discharged into the atmosphere for which a standard is applicable. The Administrator shall use the following to determine emission rate:

(1) Emission factors as specified in the latest issue of "Compilation of Air Pollutant Emission Factors," EPA Publication No. AP-42, or other emission factors determined by the Administrator to be superior to AP-42 emission factors, in cases where utilization of emission factors demonstrate that the emission level resulting from the physical or operational change will either clearly increase or clearly not increase.

(2) Material balances, continuous monitor data, or manual emission tests in cases where utilization of emission factors as referenced in paragraph (b)(1) of this section does not demonstrate to the Administrator's satisfaction whether the emission level resulting from the physical or operational change will either clearly increase or clearly not increase, or where an owner or operator demonstrates to the Administrator's satisfaction that there are reasonable grounds to dispute the result obtained by the Administrator utilizing emission factors as referenced in paragraph (b)(1) of this section. When the emission rate is based on results from manual emission tests or continuous monitoring systems, the procedures specified in Appendix C of this part shall be used to determine whether an increase in emission rate

has occurred. Tests shall be conducted under such conditions as the Administrator shall specify to the owner or operator based on representative performance of the facility. At least three valid test runs must be conducted before and at least three after the physical or operational change. All operating parameters which may affect emissions must be held constant to the maximum feasible degree for all test runs.

(c) The addition of an affected facility to a stationary source as an expansion to that source or as a replacement for an existing facility shall not by itself bring within the applicability of this part any other facility within that source.

(d) [Reserved]

(e) The following shall not, by themselves, be considered modifications under this part:

(1) Maintenance, repair, and replacement which the Administrator determines to be routine for a source category, subject to the provisions of paragraph (c) of this section and § 60.15.

(2) An increase in production rate of an existing facility, if that increase can be accomplished without a capital expenditure on that facility.

(3) An increase in the hours of operation.

(4) Use of an alternative fuel or raw material if, prior to the date any standard under this part becomes applicable to that source type, as provided by § 60.1, the existing facility was designed to accommodate that alternative use. A facility shall be considered to be designed to accommodate an alternative fuel or raw material if that use could be accomplished under the facility's construction specifications as amended prior to the change. Conversion to coal required for energy considerations, as specified in section 111(a)(8) of the Act, shall not be considered a modification.

(5) The addition or use of any system or device whose primary function is the reduction of air pollutants, except when an emission control system is removed or is replaced by a system which the Administrator determines to be less environmentally beneficial.

(6) The relocation or change in ownership of an existing facility.

(f) Special provisions set forth under an applicable subpart of this part shall supersede any conflicting provisions of this section.

(g) Within 180 days of the completion of any physical or operational change subject to the control measures specified in paragraph (a) of this section, compliance with all applicable standards must be achieved.

[40 FR 58419, Dec. 16, 1975, amended at 43 FR 34347, Aug. 3, 1978; 45 FR 5617, Jan. 23, 1980]

§ 60.15 Reconstruction.

(a) An existing facility, upon reconstruction, becomes an affected facility, irrespective of any change in emission rate.

(b) "Reconstruction" means the replacement of components of an existing facility to such an extent that:

(1) The fixed capital cost of the new components exceeds 50 percent of the fixed capital cost that would be required to construct a comparable entirely new facility, and

(2) It is technologically and economically feasible to meet the applicable standards set forth in this part.

(c) "Fixed capital cost" means the capital needed to provide all the depreciable components.

(d) If an owner or operator of an existing facility proposes to replace components, and the fixed capital cost of the new components exceeds 50 percent of the fixed capital cost that would be required to construct a comparable entirely new facility, he shall notify the Administrator of the proposed replacements. The notice must be postmarked 60 days (or as soon as practicable) before construction of the replacements is commenced and must include the following information:

(1) Name and address of the owner or operator.

(2) The location of the existing facility.

(3) A brief description of the existing facility and the components which are to be replaced.

(4) A description of the existing air pollution control equipment and the

proposed air pollution control equipment.

(5) An estimate of the fixed capital cost of the replacements and of constructing a comparable entirely new facility.

(6) The estimated life of the existing facility after the replacements.

(7) A discussion of any economic or technical limitations the facility may have in complying with the applicable standards of performance after the proposed replacements.

(e) The Administrator will determine, within 30 days of the receipt of the notice required by paragraph (d) of this section and any additional information he may reasonably require, whether the proposed replacement constitutes reconstruction.

(f) The Administrator's determination under paragraph (e) shall be based on:

(1) The fixed capital cost of the replacements in comparison to the fixed capital cost that would be required to construct a comparable entirely new facility;

(2) The estimated life of the facility after the replacements compared to the life of a comparable entirely new facility;

(3) The extent to which the components being replaced cause or contribute to the emissions from the facility; and

(4) Any economic or technical limitations on compliance with applicable standards of performance which are inherent in the proposed replacements.

(g) Individual subparts of this part may include specific provisions which refine and delimit the concept of reconstruction set forth in this section.

[40 FR 58420, Dec. 16, 1975]

§ 60.16 Priority list.

PRIORITIZED MAJOR SOURCE CATEGORIES

Priority Number[*]

Source Category

1. Synthetic Organic Chemical Manufacturing
   (a) Unit processes
   (b) Storage and handling equipment
   (c) Fugitive emission sources
   (d) Secondary sources
2. Industrial Surface Coating: Cans
3. Petroleum Refineries: Fugitive Sources
4. Industrial Surface Coating: Paper
5. Dry Cleaning
   (a) Perchloroethylene
   (b) Petroleum solvent
6. Graphic Arts
7. Polymers and Resins: Acrylic Resins
8. Mineral Wool
9. Stationary Internal Combustion Engines
10. Industrial Surface Coating: Fabric
11. Fossil-Fuel-Fired Steam Generators: Industrial Boilers
12. Incineration: Non-Municipal
13. Non-Metallic Mineral Processing
14. Metallic Mineral Processing
15. Secondary Copper
16. Phosphate Rock Preparation
17. Foundries: Steel and Gray Iron
18. Polymers and Resins: Polyethylene
19. Charcoal Production
20. Synthetic Rubber
    (a) Tire manufacture
    (b) SBR production
21. Vegetable Oil
22. Industrial Surface Coating: Metal Coil
23. Petroleum Transportation and Marketing
24. By-Product Coke Ovens
25. Synthetic Fibers
26. Plywood Manufacture
27. Industrial Surface Coating: Automobiles
28. Industrial Surface Coating: Large Appliances
29. Crude Oil and Natural Gas Production
30. Secondary Aluminum
31. Potash
32. Sintering: Clay and Fly Ash
33. Glass
34. Gypsum
35. Sodium Carbonate
36. Secondary Zinc
37. Polymers and Resins: Phenolic
38. Polymers and Resins: Urea—Melamine
39. Ammonia
40. Polymers and Resins: Polystyrene
41. Polymers and Resins: ABS-SAN Resins
42. Fiberglass
43. Polymers and Resins: Polypropylene
44. Textile Processing
45. Asphalt Roofing Plants
46. Brick and Related Clay Products
47. Ceramic Clay Manufacturing
48. Ammonium Nitrate Fertilizer
49. Castable Refractories
50. Borax and Boric Acid
51. Polymers and Resins: Polyester Resins
52. Ammonium Sulfate
53. Starch
54. Perlite
55. Phosphoric Acid: Thermal Process
56. Uranium Refining
57. Animal Feed Defluorination

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[*] Low numbers have highest priority; e.g., No. 1 is high priority, No. 59 is low priority.

58. Urea (for fertilizer and polymers)
59. Detergent

OTHER SOURCE CATEGORIES

Lead acid battery manufacture**
Organic solvent cleaning**
Industrial surface coating: metal furniture**
Stationary gas turbines***

**Mincr source category, but included on list since an NSPS is being developed for that source category.

***Not prioritized, since an NSPS for this major source category has already been proposed.

[44 FR 49225, Aug. 21, 1979]

APPENDIX B

EPA METHODS 1, 2, 3, AND 7 AND PERFORMANCE SPECIFICATION 2

METHOD 1.—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m^2 (113 $in.^2$) cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

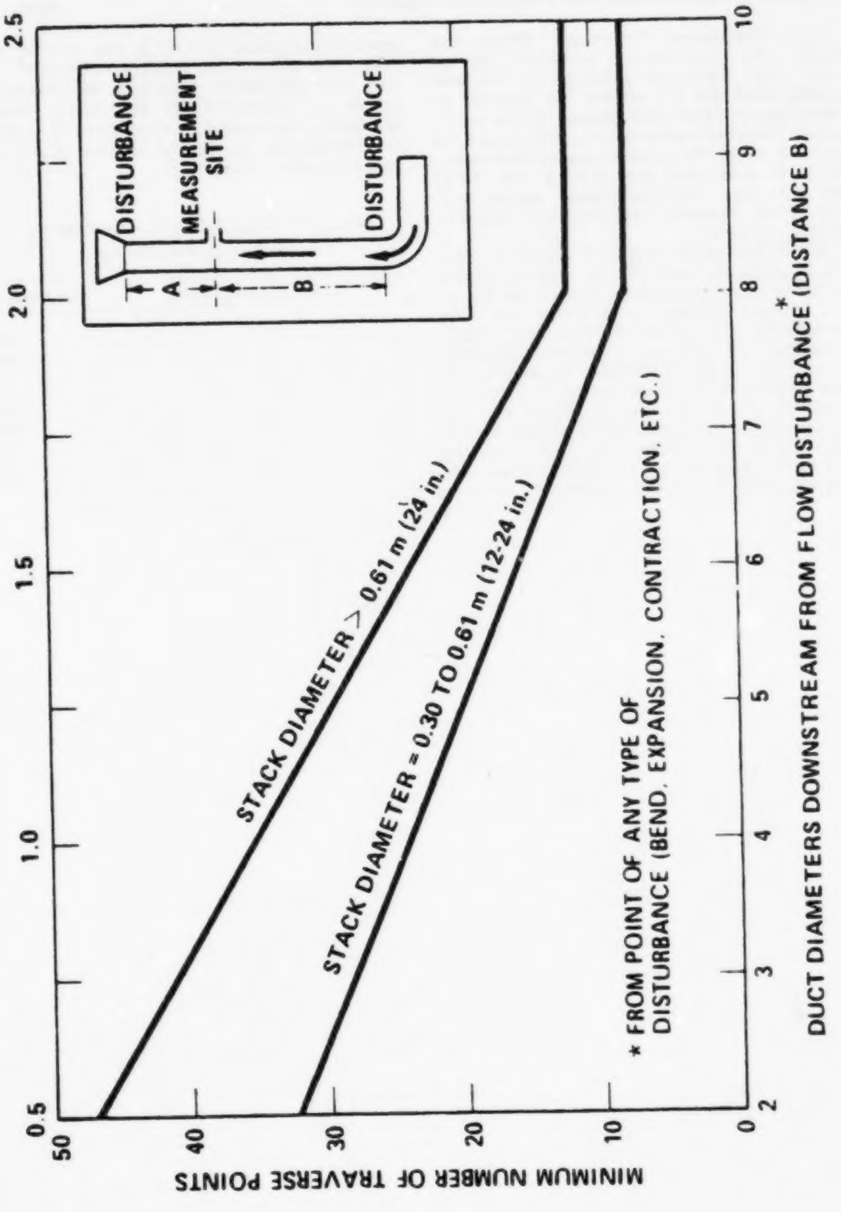
2. *Procedure*

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_e) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2\,LW}{(L+W)}$$

where L=length and W=width.

2.2 Determining the Number of Traverse Points.


0.5
1.0
1.5
2.0
2.5
50
40
30
20
10
0
2
3
4
5
6
7
8
9
10
MINIMUM NUMBER OF TRAVERSE POINTS
DUCT DIAMETERS DOWNSTREAM FROM FLOW DISTURBANCE* (DISTANCE B)
STACK DIAMETER > 0.61 m (24 in.)
STACK DIAMETER = 0.30 TO 0.61 m (12-24 in.)
* FROM POINT OF ANY TYPE OF DISTURBANCE (BEND, EXPANSION, CONTRACTION, ETC.)
DISTURBANCE
MEASUREMENT SITE
DISTURBANCE
A
B

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

TABLE 1-1. CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

Number of traverse points	Matrix layout
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

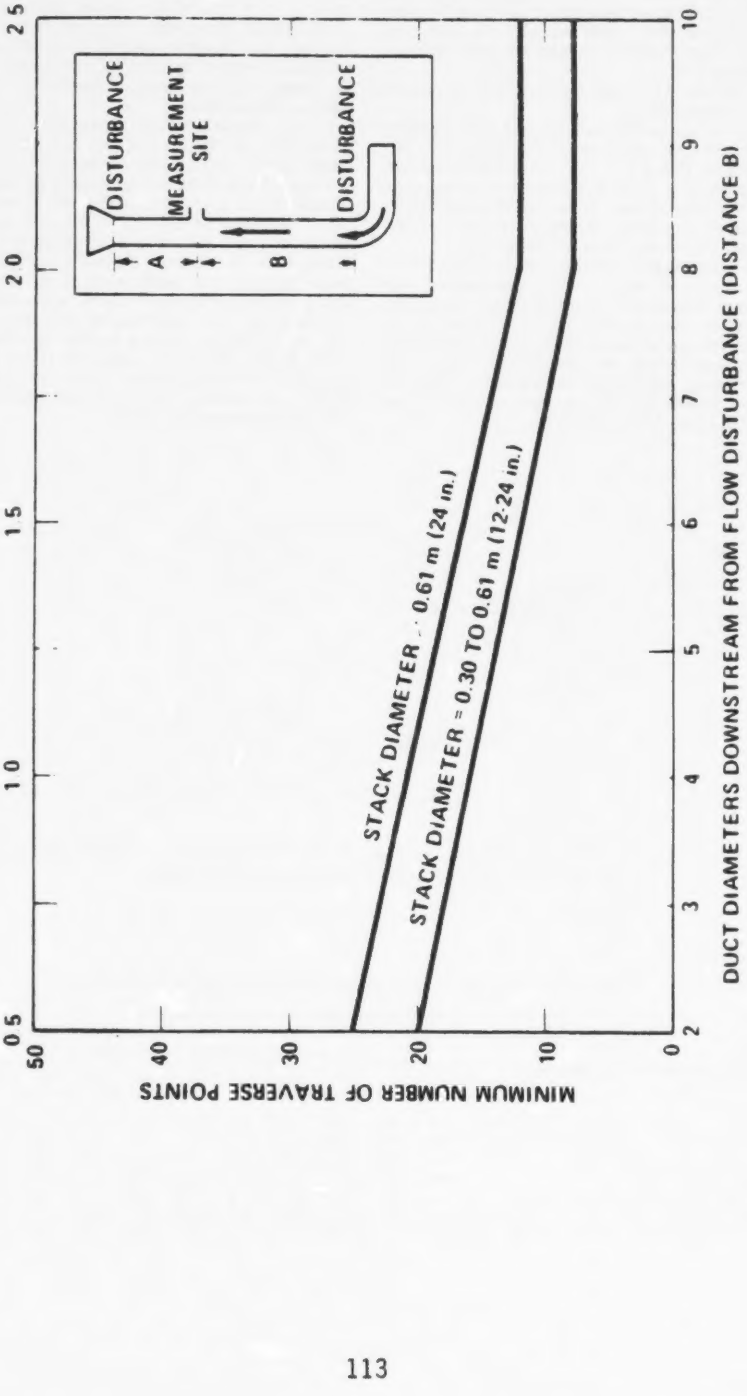


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

TRAVERSE POINT	DISTANCE, % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

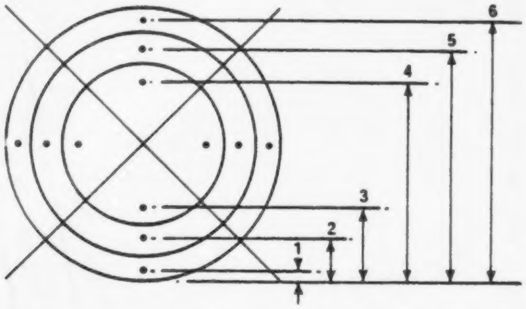


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

TABLE 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

[Percent of stack diameter from inside wall to traverse point]

Traverse point number on a diameter	Number of traverse points on a diameter—											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1

TABLE 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS—Continued

[Percent of stack diameter from inside wall to traverse point]

Traverse point number on a diameter	Number of traverse points on a diameter—											
	2	4	6	8	10	12	14	16	18	20	22	24
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4x3 "minimum number of points; matrix were expanded to 36 points, the final matrix could be 9x4 or 12x3, and would not necessarily have to be 6x6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

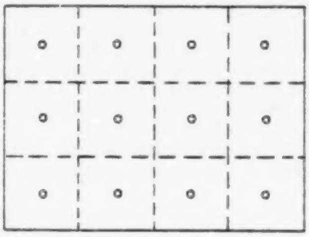


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to ±90° yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α; assign α values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of α is greater than 10°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

3. *Bibliography*

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6. Entropy Environmentalists, Inc. Determination of the Optimum Number of Sampling Points: An Analysis of Method 1 Criteria. Environmental Protection Agency, Research Triangle Park, N.C. EPA Contract No. 68-01-3172, Task 7.

METHOD 2—DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. *Principle and Applicability*

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. *Apparatus*

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

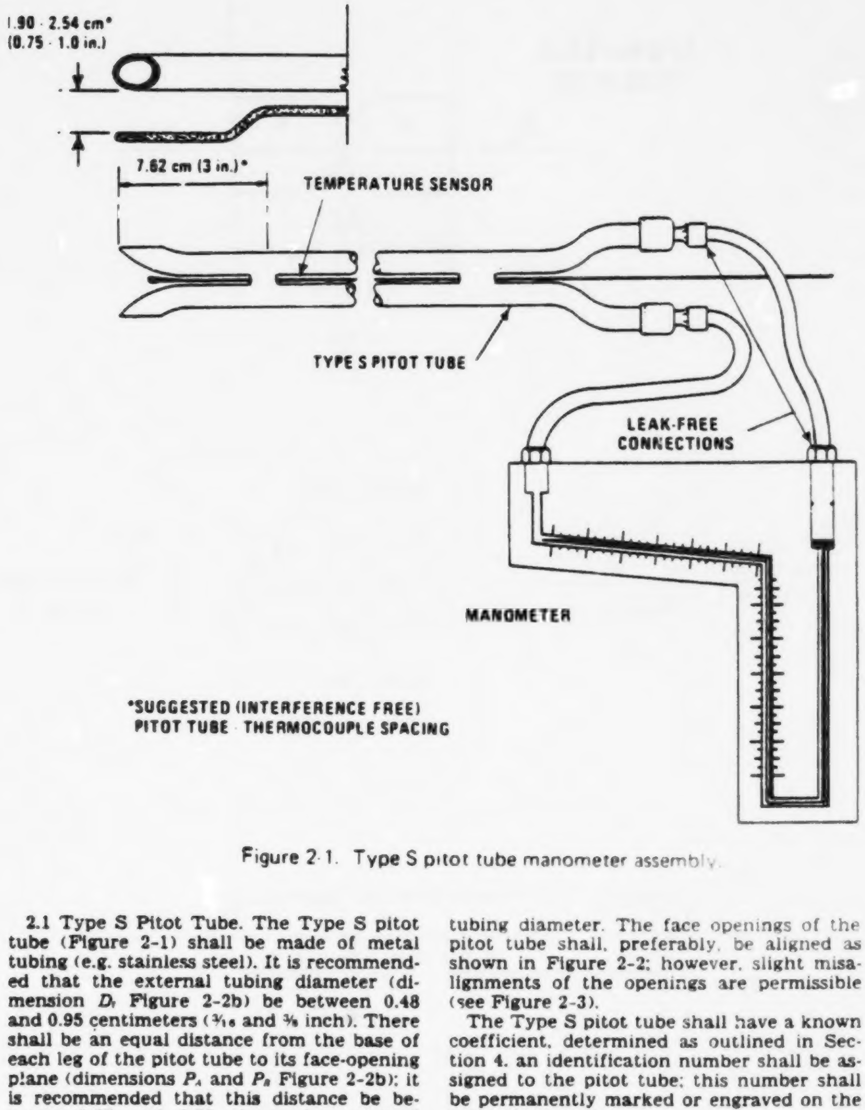

1.90 - 2.54 cm*
(0.75 - 1.0 in.)
7.62 cm (3 in.)*
TEMPERATURE SENSOR
TYPE S PITOT TUBE
LEAK-FREE CONNECTIONS
MANOMETER
*SUGGESTED (INTERFERENCE FREE)
PITOT TUBE - THERMOCOUPLE SPACING

Figure 2-1. Type S pitot tube manometer assembly.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g. stainless steel). It is recommended that the external tubing diameter (dimension D_t Figure 2-2b) be between 0.48 and 0.95 centimeters ($^3/_{16}$ and $^3/_8$ inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_A and P_B Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. an identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

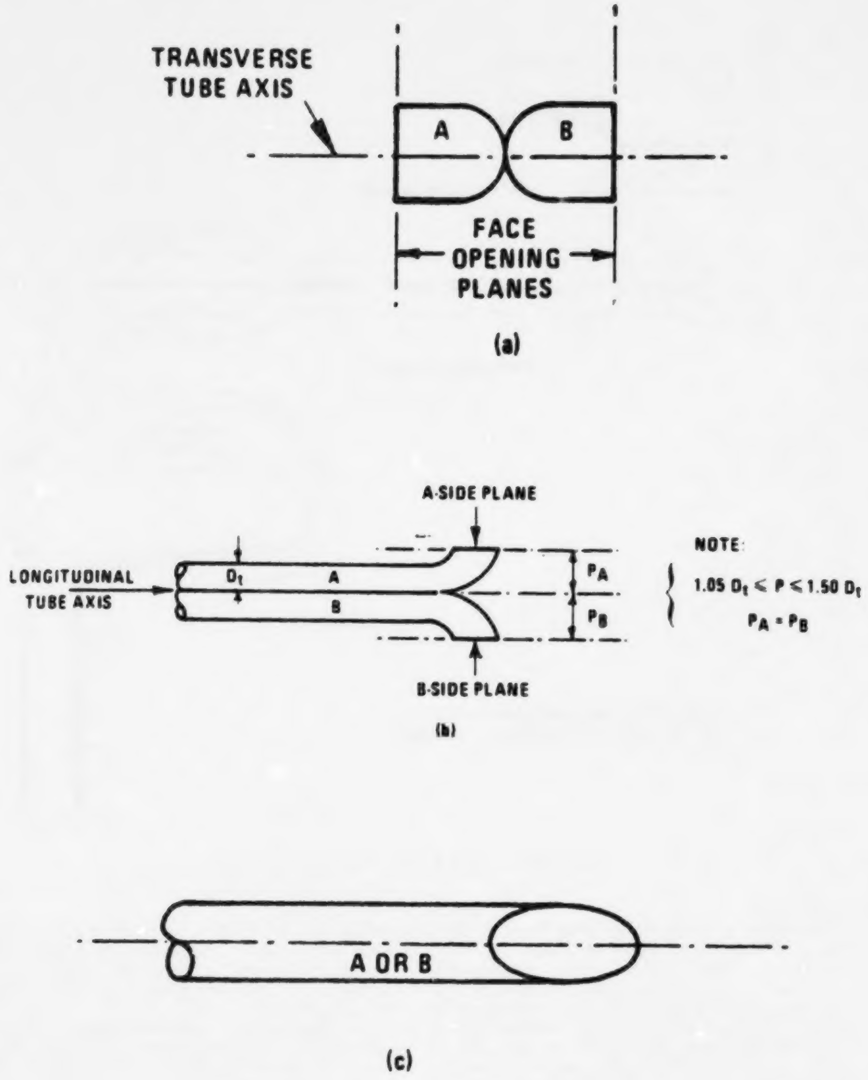
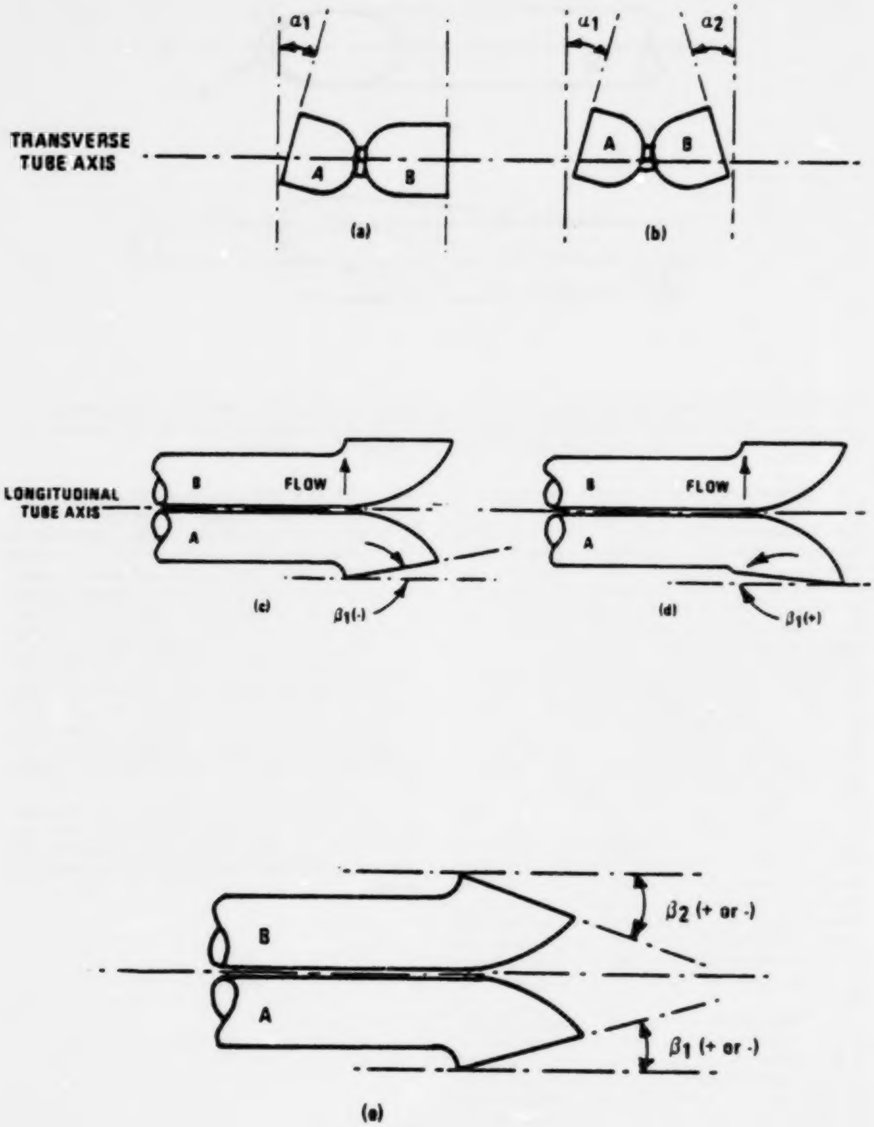

TRANSVERSE TUBE AXIS
A
B
FACE OPENING PLANES
(a)
A-SIDE PLANE
LONGITUDINAL TUBE AXIS
D_t
A
B
P_A
P_B
B-SIDE PLANE
NOTE:
1.05 D_t ≤ P ≤ 1.50 D_t
P_A = P_B
(b)
A OR B
(c)

Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.


α1
α1
α2
TRANSVERSE TUBE AXIS
A
B
A
B
(a)
(b)
LONGITUDINAL TUBE AXIS
B
FLOW
A
B
FLOW
A
(c)
β1(-)
(d)
β1(+)
β2 (+ or -)
B
A
β1 (+ or -)
(e)

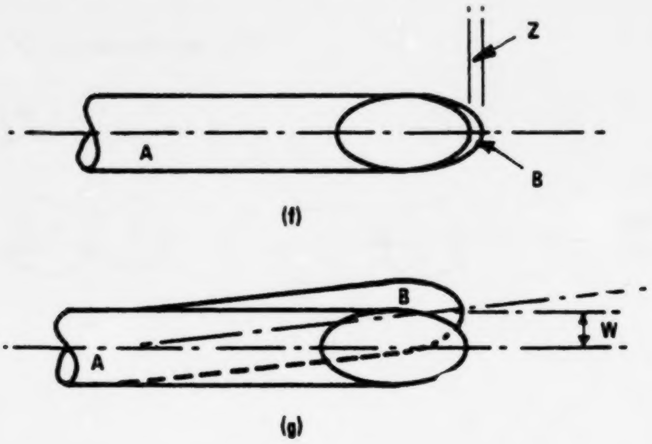


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of Cp(s) so long as $\alpha 1$ and $\alpha 2$ 10°, $\beta 1$ and $\beta 2$ 5°, z 0.32 cm (1/8 in.) and w 0.08 cm (1/32 in.) (citation 11 in Section 6).

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (±5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative Δp readings shall be taken, as above, for the last two back purges at which suitably high Δp readings are observed.

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H_2O divisions on the 0-to 1-in. inclined scale, and 0.1-in. H_2O divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H_2O. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H_2O; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H_2O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H_2O. Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T=\frac{\sum_{i=1}^{n}\sqrt{\Delta p_i+K}}{\sum_{i=1}^{n}\sqrt{\Delta p_i}}$$

where:

Δp_i = Individual velocity head reading at a traverse point, mm H_2O (in. H_2O).

n = Total number of traverse points.

$K = 0.13$ mm H_2O when metric units are used and 0.005 in H_2O when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE: If differential pressure gauges other than inclined manometers are used (e.g., magnehelic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge, capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pilot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01.

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D, the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately 0.1 D), equally spaced in a piezometer ring configuration.

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in H_2O) for Δp values between 1.3 and 25 mm H_2O (0.05 and 1.0 in. H_2O), and to the nearest 1.3 mm H_2O (0.05 in. H_2O) for Δp values above 25 mm H_2O (1.0 in. H_2O). A special, more sensitive gauge will be required to read Δp values below 1.3 mm H_2O [0.05 in. H_2O] (see Citation 18 in Section 6).

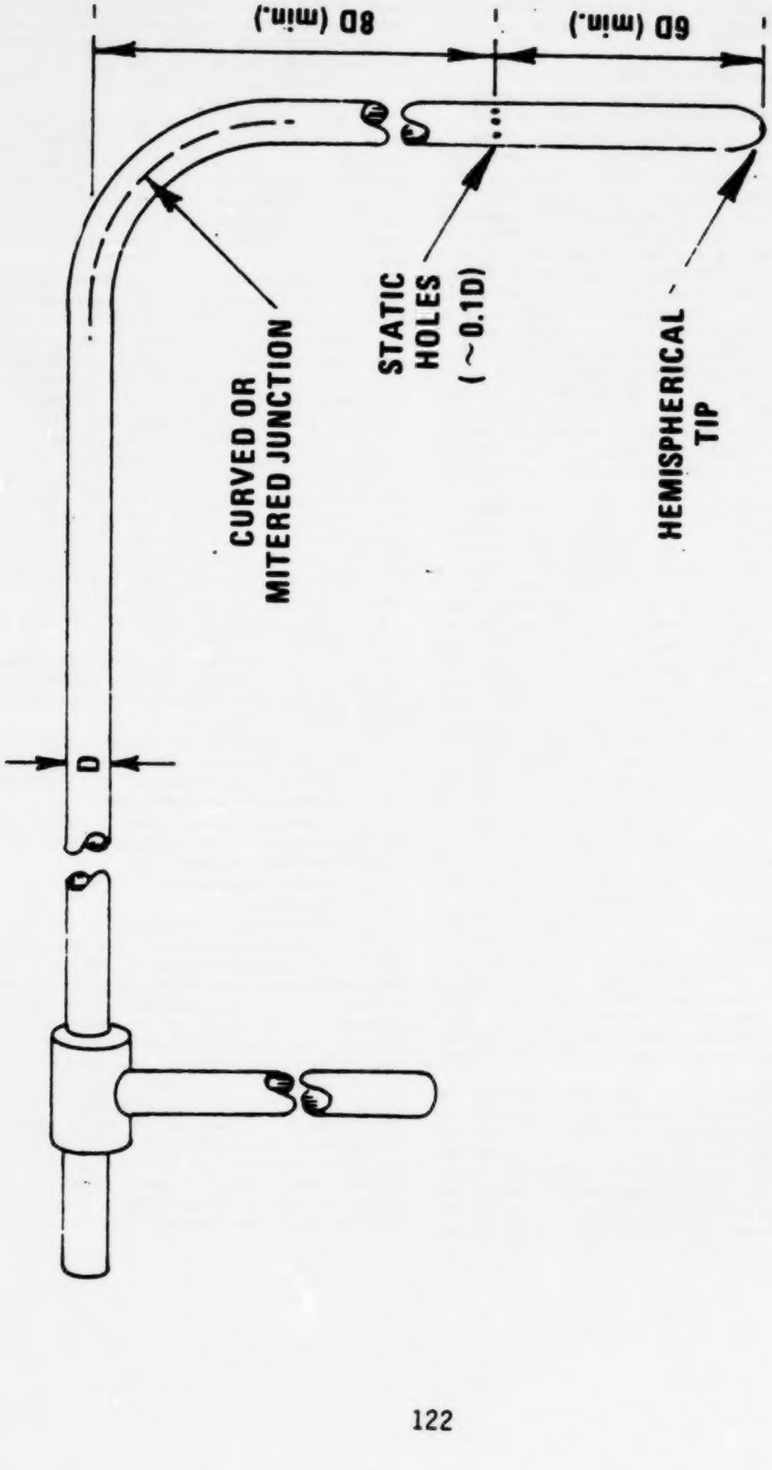

8D (min.)
6D (min.)
D
CURVED OR MITERED JUNCTION
STATIC HOLES (~0.1D)
HEMISPHERICAL TIP

Figure 2-4. Standard pitot tube design specifications.

3. *Procedure*

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H_2O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H_2O. Other leak-check procedures, subject to the approval of the Administrator may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

PLANT ______________________________
DATE ________________ RUN NO. ____________
STACK DIAMETER OR DIMENSIONS, m(in.) ____________
BAROMETRIC PRESSURE, mm Hg (in. Hg) ____________
CROSS SECTIONAL AREA, m^2(ft^2) ____________
OPERATORS ______________________________
PITOT TUBE I.D. NO. ______________________________
AVG. COEFFICIENT, C_p = ______________________________
LAST DATE CALIBRATED ______________________________

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack Temperature		P_g mm Hg (in.Hg)	$\sqrt{\Delta p}$
		t_s, °C (°F)	T_s, °K (°R)		
		Average			

Figure 2-5. Velocity traverse data.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO_2, O_2, CO, and N_2, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4. *Calibration*

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

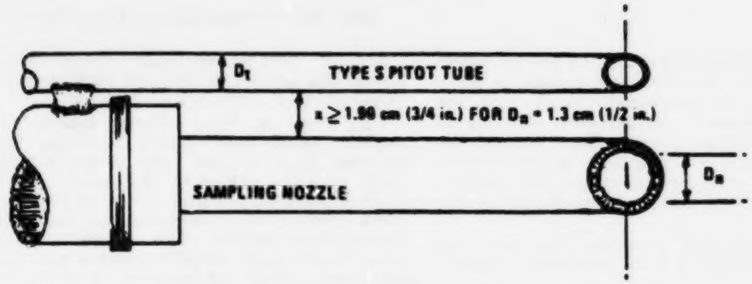
After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension D_t, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2-2b). If D_t is between 0.48 and 0.95 cm (3/16 and 3/8 in.) and if P_A and P_B are equal and between 1.05 and 1.50 D_t, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1).

If D_t, P_A, and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

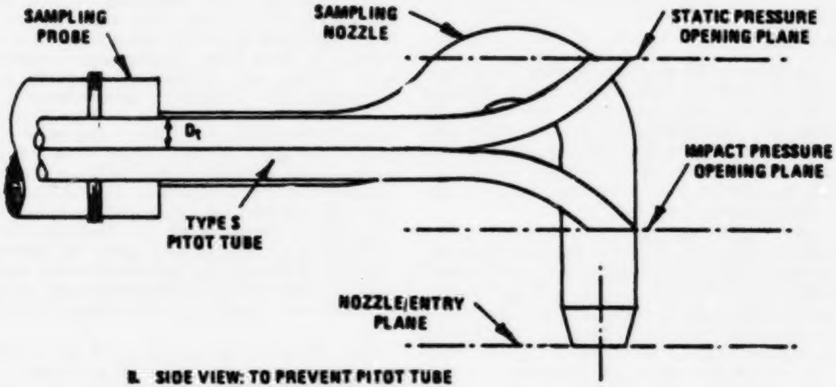
4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6); therefore an assigned (or otherwise known) baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

NOTE: Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:


D_t
TYPE S PITOT TUBE
$x \geq 1.90$ cm (3/4 in.) FOR $D_n = 1.3$ cm (1/2 in.)
SAMPLING NOZZLE
D_n

A. BOTTOM VIEW; SHOWING MINIMUM PITOT-NOZZLE SEPARATION.


SAMPLING PROBE
SAMPLING NOZZLE
STATIC PRESSURE OPENING PLANE
D_t
IMPACT PRESSURE OPENING PLANE
TYPE S PITOT TUBE
NOZZLE/ENTRY PLANE

B. SIDE VIEW: TO PREVENT PITOT TUBE FROM INTERFERING WITH GAS FLOW STREAMLINES APPROACHING THE NOZZLE, THE IMPACT PRESSURE OPENING PLANE OF THE PITOT TUBE SHALL BE EVEN WITH OR ABOVE THE NOZZLE ENTRY PLANE.

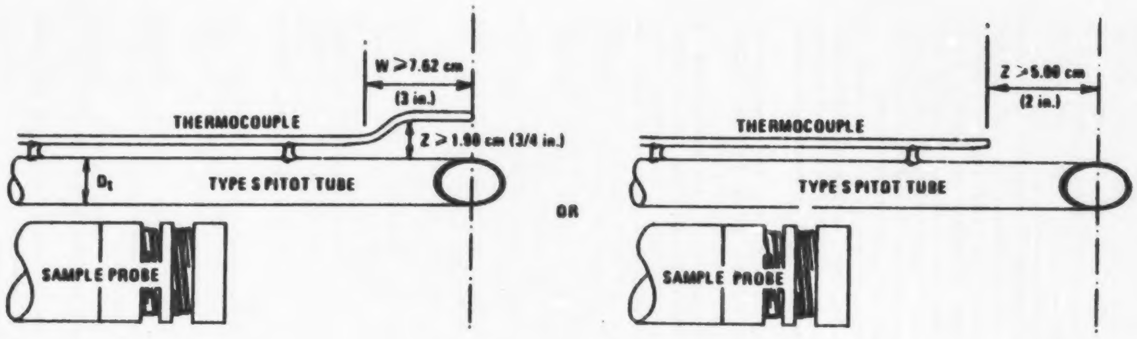

THERMOCOUPLE
W ≥7.62 cm
(3 in.)
Z ≥1.90 cm (3/4 in.)
D_t
TYPE S PITOT TUBE
SAMPLE PROBE
OR
THERMOCOUPLE
Z ≥5.08 cm
(2 in.)
TYPE S PITOT TUBE
SAMPLE PROBE

Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

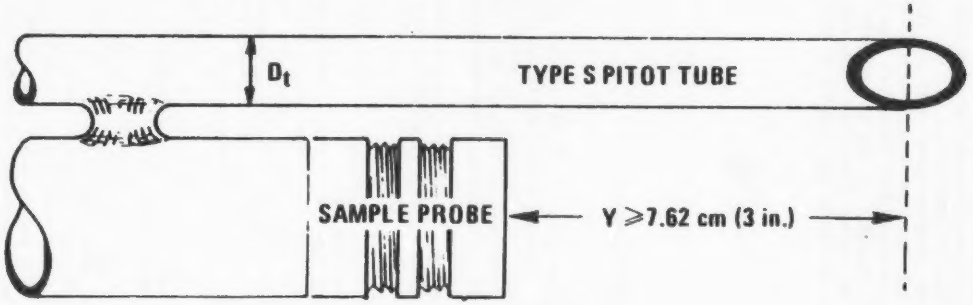

D_t
TYPE S PITOT TUBE
SAMPLE PROBE
Y ≥7.62 cm (3 in.)

Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2LW}{(L+W)}$$

Equation 2-1

where:
D_e = Equivalent diameter
L = Length
W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

NOTE: The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 ft/min) will generally be valid to within ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to within ±5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 ft/min). If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular car should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read Δp_{std} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_s and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coeffficient as follo

PITOT TUBE IDENTIFICATION NUMBER: ______________ DATE: ____________

CALIBRATED BY: ____

	"A" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(A)$
1				
2				
3				
		$\bar{C}_p$ (SIDE A)		

	"B" SIDE CALIBRATION			
RUN NO.	Δp_{std} cm H_2O (in. H_2O)	$\Delta p_{(s)}$ cm H_2O (in. H_2O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(B)$
1				
2				
3				
		$\bar{C}_p$ (SIDE B)		

$$\text{AVERAGE DEVIATION} = \sigma\,(\text{A OR B}) = \frac{\sum_{1}^{3} \left| C_{p(s)} - \bar{C}_p(\text{A OR B}) \right|}{3} \longleftarrow \text{MUST BE} \leq 0.01$$

$$\left| \bar{C}_p\,(\text{SIDE A}) - \bar{C}_p\,(\text{SIDE B}) \right| \longleftarrow \text{MUST BE} \leq 0.01$$

Figure 2-9. Pitot tube calibration data.

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Equation 2-2

where:

$C_{p(s)}$ = Type S pitot tube coefficient

$C_{p(std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.

Δp_{std} = Velocity head measured by the standard pitot tube, cm H_2O (in. H_2O)

Δp_s = Velocity head measured by the Type S pitot tube, cm H_2O (in H_2O)

4.1.4.2 Calculate C_p (side A), the mean A-side coefficient, and C_p (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from C_p (side A), and the deviation of each B-side value of $C_{p(s)}$ from C_p C_p (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \overline{C}_p(\text{A or B})$$

Equation 2-3

4.1.4.4 Calculate δ, the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma\,(\text{side A or B}) = \frac{\sum_{1}^{3} |C_{p(s)} - \overline{C}_p(A \text{ or } B)|}{3}$$

Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the values of δ (side A) and δ (side B) are less than or equal to 0.01 and if the absolute value of the difference between C_p (A) and C_p (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

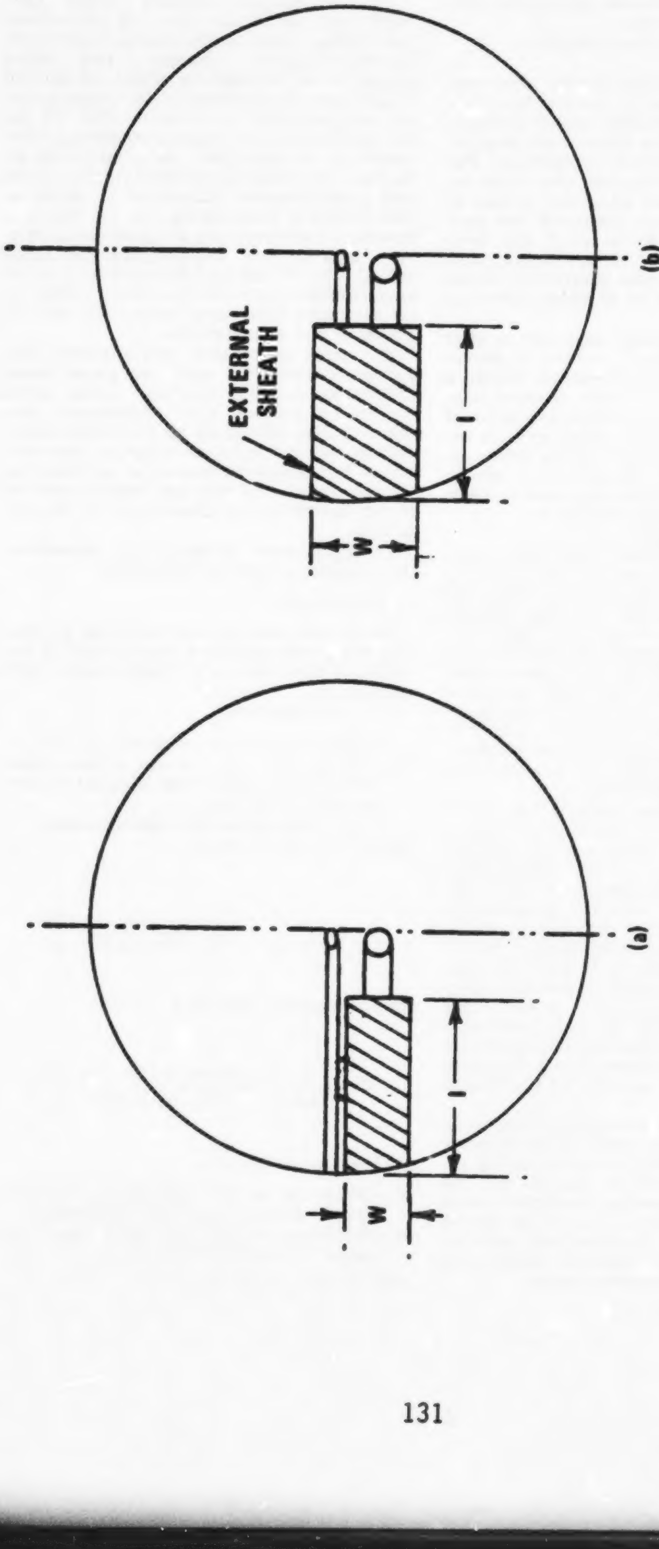
4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., C_p (side A) and C_p (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6, and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or ¼ in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (δ) value of 0.01 or less (see Section 4.1.4.4).


EXTERNAL SHEATH
l
W
(b)
l
W
(a)

$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times W}{\text{DUCT AREA}}\right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

Figure 2-10. Projected-area models for typical pitot tube assemblies.

4.1.6 Field Use and Recalibration.

4.1.6.1 Field Use.

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of $C_{p(s)}$. Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration.

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damanged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405° C (761° F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405° C (761° F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

4.4 Barometer. Calibrate the barometer used against a mercury barometer.

5. *Calculations*

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

5.1 Nomenclature.

A = Cross-sectional area of stack, m² (ft²).

B_{ws} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

C_p = Pitot tube coefficient, dimensionless.

K_p = Pitot tube constant,

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(g/g\text{-mole})(\text{mm Hg})}{(^\circ\text{K})(\text{mm H}_2\text{O})} \right]^{1/2}$$

for the metric system and

$$85.49 \frac{\text{ft}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(^\circ\text{R})(\text{in. H}_2\text{O})} \right]^{1/2}$$

for the English system.

M_d = Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).

M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$= M_d (1 - B_{ws}) + 18.0\, B_{ws}$

Equation 2-5

P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).

P_g = Stack static pressure, mm Hg (in. Hg).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

$= P_{bar} + P_g$

Equation 2-6

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

t_s = Stack temperature, °C (°F).

T_s = Absolute stack temperature, °K. (°R).

$= 273 + t_s$ for metric

Equation 2-7

$= 460 + t_s$ for English

Equation 2-8

T_{std} = Standard absolute temperature, 293 °K (528° R)

v_s = Average stack gas velocity, m/sec (ft/sec).

Δ_p = Velocity head of stack gas, mm H_2O (in. H_2O).

3,600 = Conversion factor, sec/hr.

18.0 = Molecular weight of water, g/g-mole (lb/lb-mole).

5.2 Average stack gas velocity.

$$v_s = K_p C_p (\sqrt{\Delta p})_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

Equation 2-9

5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3{,}600\ (1 - B_{ws}) v_s A \left(\frac{T_{std}}{T_{s(avg)}}\right)\left(\frac{P_s}{P_{std}}\right)$$

Equation 2-10

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METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. *Principle and Applicability*

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO_2), percent oxygen (O_2), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be made, either an Orsat or a Fyrite[1] analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO_2 and O_2 concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO_2, O_2, CO, and nitrogen (N_2) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO_2 or O_2 and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency.

2. *Apparatus*

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1).

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other materials inert to O_2, CO_2, CO, and N_2 and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2).

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

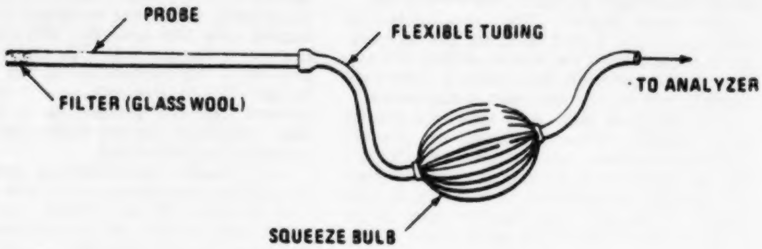
PROBE
FLEXIBLE TUBING
TO ANALYZER
FILTER (GLASS WOOL)
SQUEEZE BULB

Figure 3-1. Grab-sampling train.

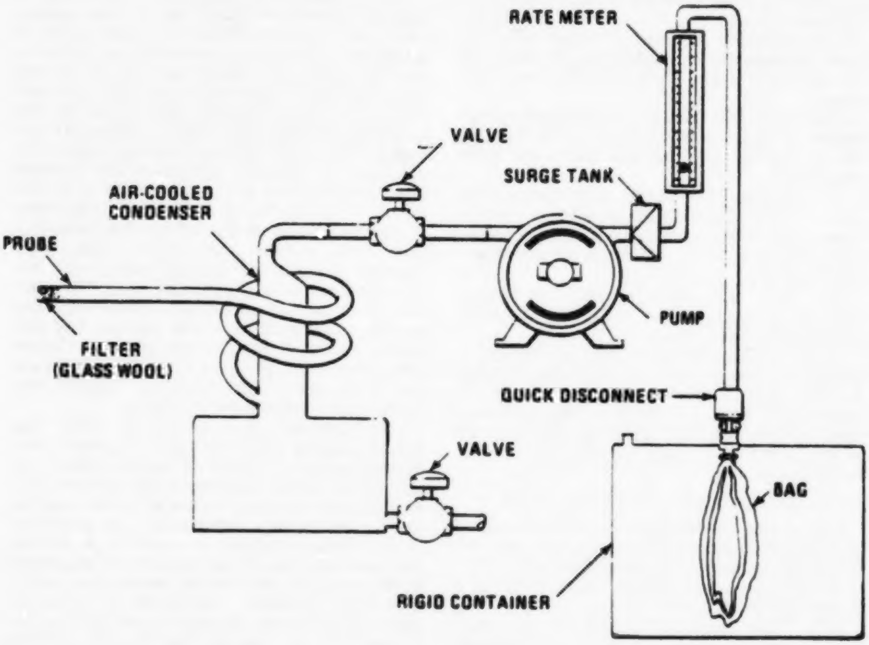
RATE METER
VALVE
SURGE TANK
AIR-COOLED CONDENSER
PROBE
PUMP
FILTER (GLASS WOOL)
QUICK DISCONNECT
VALVE
BAG
RIGID CONTAINER

Figure 3-2. Integrated gas-sampling train.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2, CO_2, CO, and N_2, may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ±2 percent of the selected flow rate. A flow rate range of 500 to 1000 cm^3/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 55 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 28 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater that 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. *Dry Molecular Weight Determination*

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If and Orsat analyzer is used, it is recommended that the analyzer be leaked-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (lb/lb-mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft^3) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite-type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in sections 3.2.2 throught 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

4. *Emission Rate Correction Factor or Excess Air Determination*

NOTE: A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

TIME	TRAVERSE PT.	Q lpm	% DEV.[a]
	AVERAGE		

$$^{a}\%\ \text{DEV} = \left(\frac{Q - Q\ \text{avg}}{Q\ \text{avg}}\right) 100 \qquad (\text{MUST BE} \leqslant 10\%)$$

Figure 3-3. Sampling rate data.

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis.

Note: Since this single-point, grab sampling and analytical procedure in normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of a least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total lengh of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft^3) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-check (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To insure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three of four) should be make between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more that (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2 and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before an after the analysis.

Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

5. *Leak-Check Procedure for Orsat Analyzers*

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be throughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.1.3 Record the meniscus position.

5.1.4 Observe the menicus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

5.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. *Calculations*

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).
%EA = Percent excess air.
$\%CO_2$ = Percent CO_2 by volume (dry basis).
$\%O_2$ = Percent O_2 by volume (dry basis).
%CO = Percent CO by volume (dry basis).
$\%N_2$ = Percent N_2 by volume (dry basis).
0.264 = Ratio of O_2 to N_2 in air, v/v.
0.280 = Molecular weight of N_2 or CO, divided by 100.
0.320 = Molecular weight of O_2 divided by 100.
0.440 = Molecular weight of CO_2 divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable), by substituting the appropriate values of perrent O_2, CO, and N_2 (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA=\left[\frac{\%O_2-0.5\%CO}{0.264\%N_2(\%O_2-0.5\%CO)}\right]100$$

Equation 3-1

NOTE: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas

$$M_d=0.440(\%CO_2)+0.320(\%O_2)+0.280(\%N_2+\%CO)$$

Equation 3-2

NOTE: The above equation does not consider argon in air (about 0.9 percent, moleculars weight of 37.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

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METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

1. *Principle and Applicability*

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimeterically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO_x (as NO_2) per dry standard cubic meter, without having to dilute the sample.

2. *Apparatus*

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon[3] tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

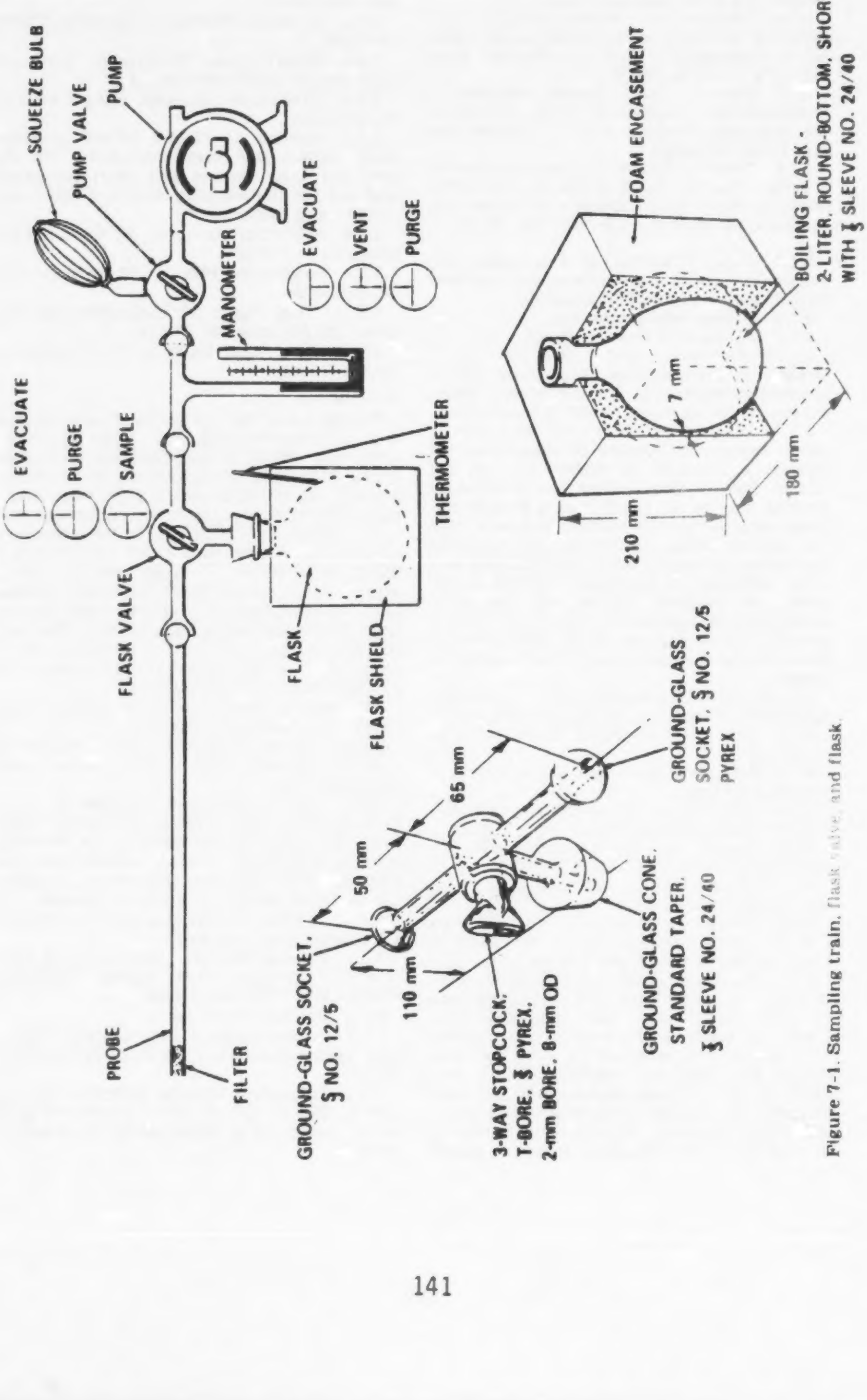
PROBE
FILTER
FLASK VALVE
FLASK
FLASK SHIELD
THERMOMETER
EVACUATE
PURGE
SAMPLE
MANOMETER
SQUEEZE BULB
PUMP VALVE
PUMP
EVACUATE
VENT
PURGE
GROUND-GLASS SOCKET, § NO. 12/5
110 mm
50 mm
65 mm
3-WAY STOPCOCK; T-BORE, § PYREX, 2-mm BORE, 8-mm OD
GROUND-GLASS CONE, STANDARD TAPER, § SLEEVE NO. 24/40
GROUND-GLASS SOCKET, § NO. 12/5 PYREX
FOAM ENCASEMENT
BOILING FLASK - 2-LITER, ROUND-BOTTOM, SHORT NECK, WITH § SLEEVE NO. 24/40
7 mm
210 mm
180 mm

Figure 7-1. Sampling train, flask valve, and flask.

2.1.2 Collection Flask. Two-liter borosilicate, round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask Valve. T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature Gauge. Dial-type thermometer, or other temperature gauge, capable of measuring 1° C (2° F) intervals from −5 to 50° C (25 to 125° F).

2.1.5 Vacuum Line. Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Vacuum Gauge. U-tube manometer, 1 meter (36 in.), with 1-mm (0.1-in.) divisions, or other gauge capable of measuring pressure to within ±2.5 mm Hg (0.10 in. Hg).

2.1.7 Pump. Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze Bulb. One-way.

2.1.9 Volumetric Pipette. 25 ml.

2.1.10 Stopcock and Ground Joint Grease. A high-vacuum, high-temperature chlorofluorocarbon grease is required. Halocarbon 25-5S has been found to be effective.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase, or vice versa for elevation decrease.

2.2 Sample Recovery. The following equipment is required for sample recovery:

2.2.1 Graduated Cylinder. 50 ml with 1-ml divisions.

2.2.2 Storage Containers. Leak-free polyethylene bottles.

2.2.3 Wash Bottle. Polyethylene or glass.

2.2.4 Glass Stirring Rod.

2.2.5 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3 Analysis. For the analysis, the following equipment is needed:

2.3.1 Volumetric Pipettes. Two 1 ml, two 2 ml, one 3 ml, one 4 ml, two 10 ml, and one 25 ml for each sample and standard.

2.3.2 Porcelain Evaporating Dishes. 175- to 250-ml capacity with lip for pouring, one for each sample and each standard. The Coors No. 45006 (shallow-form, 195 ml) has been found to be satisfactory. Alternatively, polymethyl pentene beakers (Nalge No. 1203, 150 ml), or glass beakers (150 ml) may be used. When glass beakers are used, etching of the beakers may cause solid matter to be present in the analytical step; the solids should be removed by filtration (see Section 4.3).

2.3.3 Steam Bath. Low-temperature ovens or thermostatically controlled hot plates kept below 70° C (160° F) are acceptable alternatives.

2.3.4 Dropping Pipette or Dropper. Three required.

2.3.5 Polyethylene Policeman. One for each sample and each standard.

2.3.6 Graduated Cylinder. 100 ml with 1-ml divisions.

2.3.7 Volumetric Flasks. 50 ml (one for each sample and each standard), 100 ml (one for each sample and each standard, and one for the working standard KNO_3 solution), and 1000 ml (one).

2.3.8 Spectrophotometer. To measure absorbance at 410 nm.

2.3.9 Graduated Pipette. 10 ml with 0.1-ml divisions.

2.3.10 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3.11 Analytical Balance. To measure to within 0.1 mg.

3. *Reagents*

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1 Sampling. To prepare the absorbing solution, cautiously add 2.8 ml concentrated H_2SO_4 to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample Recovery. Two reagents are required for sample recovery:

3.2.1 Sodium Hydroxide (1*N*). Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the $KMNO_4$ test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.3 Analysis. For the analysis, the following reagents are required:

3.3.1 Fuming Sulfuric Acid. 15 to 18 percent by weight free sulfur trioxide. HANDLE WITH CAUTION.

3.3.2 Phenol. White solid.

3.3.3 Sulfuric Acid. Concentrated, 95 percent minimum assay. HANDLE WITH CAUTION.

3.3.4 Potassium Nitrate. Dried at 105 to 110° C (220 to 230° F) for a minimum of 2 hours just prior to preparation of standard solution.

3.3.5 Standard KNO$_3$ Solution. Dissolve exactly 2.198 g of dried potassium nitrate (KNO$_3$) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1,000-ml volumetric flask.

3.3.6 Working Standard KNO$_3$ Solution. Dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One milliter of the working standard solution is equivalent to 100 μg nitrogen dioxide (NO$_2$).

3.3.7 Water. Deionized, distilled as in Section 3.2.2.

3.3.8 Phenoldisulfonic Acid Solution. Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100° C (212° F) for 2 hours. Store in a dark, stoppered bottle.

4. *Procedures*

4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free, and that all ground glass joints have been properly greased with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a pressure approaching the vapor pressure of water at the existing temperature is desirable. Turn the pump valve to its "vent" position and turn the off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve (V_f), the flask temperature (T_i), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask (V_i) is equal to the barometric pressure less the manometer reading. Immediately turn the flask valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are equal. This will usually require about 15 seconds; a longer period indicates a "plug" in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion of NO to NO$_2$ (e.g., an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in N$_2$), then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods; (1) Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg (3 in. Hg) absolute pressure or less; or (2) inject oxygen into the flask after sampling; or (3) terminate sampling with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample Recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature (T_f), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask (P_f) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH bv dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents. Seal the container for shipping.

4.3 Analysis. Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50-ml volumetric flask, and rinse the container twice with 5-ml portions of deionized, distilled water. Add the rinse water to the flask and dilute to the mark with deionized, distilled water; mix thoroughly. Pipette a 25-ml aliquot into the procelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25-ml aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Allow the solution to cool, add 20 ml deionized, distilled water, mix well by stirring, and add concentrated ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these must be removed by filtration (centrifugation is an acceptable alternative, subject to the approval of the Administrator), as follows: filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water; filter these three rinses. Wash the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are absent, the solution can be transferred directly to the 100-ml volumetric flask and diluted to the mark with deionized, distilled water. Mix the contents of the flask thoroughly, and measure the absorbance at the optimum wavelength used for the standards (Section 5.2.1), using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of deionized, distilled water if the absorbance exceeds A_4, the absorbance of the 400 μg NO_2 standard (see Section 5.2.2).

5. *Calibration*

5.1 Flask Volume. The volume of the collection flask-flask valve combination must be known prior to sampling. Assemble the flask and flask valve and fill with water, to the stopcock. Measure the volume of water to ±10 ml. Record this volume on the flask.

5.2 Spectrophotometer Calibration.

5.2.1 Optimum Wavelength Determination. Calibrate the wavelength scale of the spectrophotometer every 6 months. The calibration may be accomplished by using an energy source with an intense line emission such as a mercury lamp, or by using a series of glass filters spanning the measuring range of the spectrophotometer. Calibration materials are available commercially and from the National Bureau of Standards. Specific details on the use of such materials should be supplied by the vendor; general information about calibration techniques can be obtained from general reference books on analytical chemistry. The wavelength scale of the spectrophotometer must read correctly within ± 5 nm al all calibration points; otherwise, the specrophotometer shall be repaired and recalibrated. Once the wavelength scale of the spectrophotometer is in proper calibration, use 410 nm as the optimum wavelength for the measurement of the absorbance of the standards and samples.

Alternatively, a scanning procedure may be employed to determine the proper measuring wavelength. If the instrument is a double-beam spectrophotometer, scan the spectrum between 400 and 415 nm using a 200 μg NO_2 standard solution in the sample cell and a blank solution in the reference cell. If a peak does not occur, the spectrophotometer is probably malfunctioning and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance of both the standards and the samples. For a single-beam spectrophotometer, follow the scanning procedure described above, except that the blank and standard solutions shall be scanned separately. The optimum wavelength shall be the wavelength at which the maximum difference in absorbance between the standard and the blank occurs.

5.2.2 Determination of Spectrophotometer Calibration Factor K_c. Add 0.0 ml, 2 ml, 4 ml, 6 ml., and 8 ml of the KNO_3 working standard solution (1 ml = 100 μg NO_2) to a series of five 50-ml volumetric flasks. To each flask, add 25 ml of absorbing solution, 10 ml deionized, distilled water, and sodium hydroxide (1 N) dropwise until the pH is between 9 and 12 (about 25 to 35 drops each). Dilute to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of each solution into a separate porcelain evaporating dish. Beginning with the evaporation step, follow the analysis procedure of Section 4.3 until the solution has been transferred to the 100 ml volumetric flask and diluted to the mark. Measure the absorbance of each solution, at the optimum wavelength, as determined in Section 5.2.1. This calibration procedure must be repeated on each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

Equation 7-1

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where:

K_c = Calibration factor
A_1 = Absorbance of the 100-μg NO_2 standard
A_2 = Absorbance of the 200-μg NO_2 standard
A_3 = Absorbance of the 300-μg NO_2 standard
A_4 = Absorbance of the 400-μg NO_2 standard

5.3 Barometer. Calibrate against a mercury barometer.

5.4 Temperature Gauge. Calibrate dial thermometers against mercury-in-glass thermometers.

5.5 Vacuum Gauge. Calibrate mechanical gauges, if used, against a mercury manometer such as that specified in 2.1.6.

5.6 Analytical Balance. Calibrate against standard weights.

6. *Calculations*

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

6.1 Nomenclature.

A = Absorbance of sample.
C = Concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf).
F = Dilution factor (i.e., 25/5, 25/10, etc., required only if sample dilution was needed to reduce the absorbance into the range of calibration).
K_c = Spectrophotometer calibration factor.
m = Mass of NO_x as NO_2 in gas sample, μg.
P_f = Final absolute pressure of flask, mm Hg (in. Hg).
P_i = Initial absolute pressure of flask, mm Hg (in. Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
T_f = Final absolute temperature of flask, °K (°R).
T_i = Initial absolute temperature of flask, °K (°R).
T_{std} = Standard absolute temperature 293° K (528° R)
V_{sc} = Sample volume at standard conditions (dry basis), ml.
V_f = Volume of flask and valve, ml.
V_a = Volume of absorbing soluton, 25 ml.
2 = 50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted).

6.2 Sample volume, dry basis, corrected to standard conditions.

$$V_{sc}=\frac{T_{std}}{P_{std}}(V_f-V_a)\left[\frac{P_f}{T_f}-\frac{P_i}{T_i}\right]$$

$$=K_1(V_f-25\text{ ml})\left[\frac{P_f}{T_f}-\frac{P_i}{T_i}\right]$$

Equation 7-2

$$K_1=0.3858\ \frac{°K}{\text{mm Hg}}\text{ for metric units}$$

$$=17.64\ \frac{°R}{\text{in. Hg}}\text{ for English units}$$

6.3 Total μg NO_2 per sample.

$$m=2\,K_c\,AF$$

Equation 7-3

NOTE: If other than a 25-ml aliquot is used for analysis, the factor 2 must be replaced by a corresponding factor.

6.4 Sample concentration, dry basis, corrected to standard conditions.

$$C=K_2\frac{m}{V_{sc}}$$

Equation 7-4

where:

$$K_2=10^3\ \frac{\text{mg/m}^3}{\mu\text{g/ml}}\text{ for metric units}$$

$$=6.243\times10^{-5}\ \frac{\text{lb/scf}}{\mu\text{g/ml}}\text{ for English units}$$

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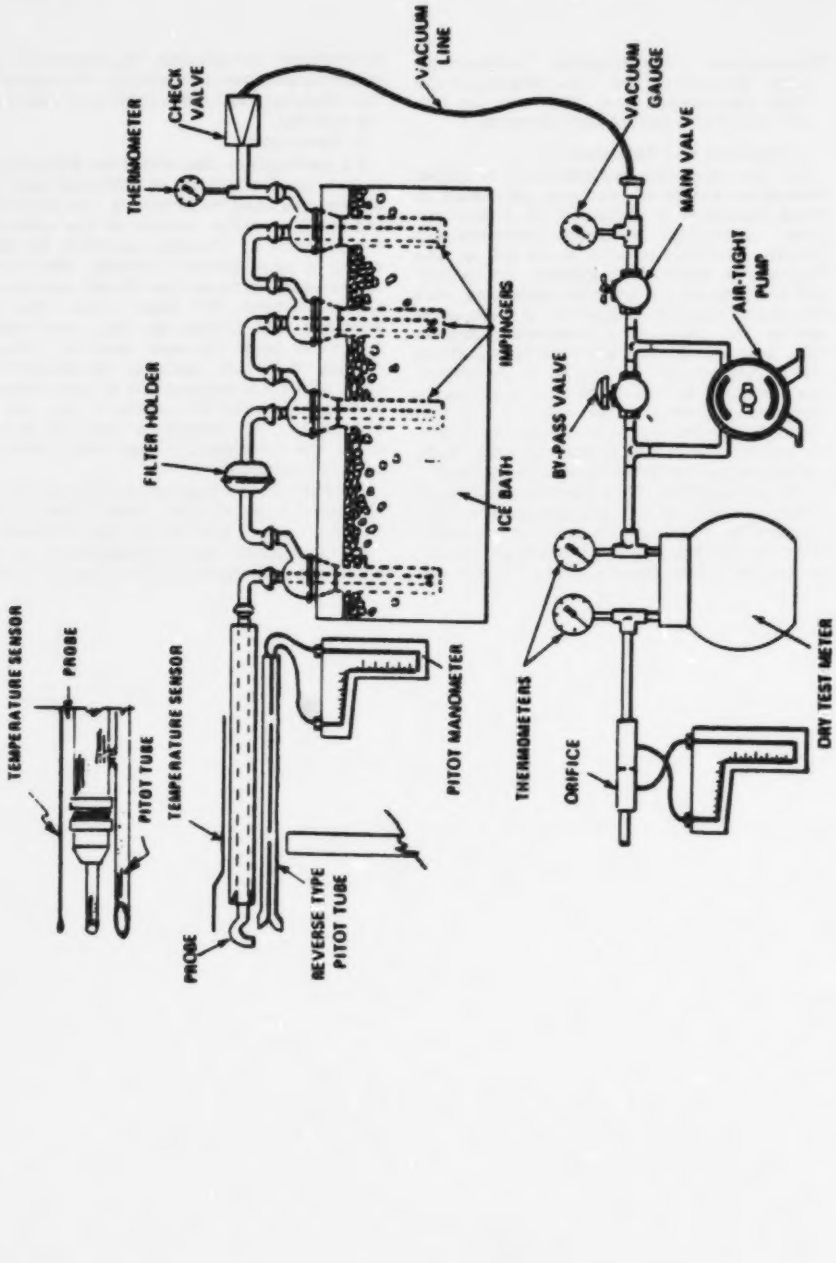

TEMPERATURE SENSOR
PROBE
PITOT TUBE
TEMPERATURE SENSOR
PROBE
REVERSE TYPE PITOT TUBE
PITOT MANOMETER
THERMOMETER
CHECK VALVE
VACUUM LINE
FILTER HOLDER
IMPINGERS
ICE BATH
VACUUM GAUGE
MAIN VALVE
AIR-TIGHT PUMP
BY-PASS VALVE
THERMOMETERS
ORIFICE
DRY TEST METER

Figure 8-1. Sulfuric acid mist sampling train

PERFORMANCE SPECIFICATION 2—PERFORMANCE SPECIFICATIONS AND SPECIFICATION TEST PROCEDURES FOR MONITORS OF SO_2 AND NO_x FROM STATIONARY SOURCES

1. Principle and Applicability.

1.1 Principle. The concentration of sulfur dioxide or oxides of nitrogen pollutants in stack emissions is measured by a continuously operating emission measurement system. Concurrent with operation of the continuous monitoring system, the pollutant concentrations are also measured with reference methods (Appendix A). An average of the continuous monitoring system data is computed for each reference method testing period and compared to determine the relative accuracy of the continuous monitoring system. Other tests of the continuous monitoring system are also performed to determine calibration error, drift, and response characteristics of the system.

1.2 Applicability. This performance specification is applicable to evaluation of continuous monitoring systems for measurement of nitrogen oxides or sulfur dioxide pollutants. These specifications contain test procedures, installation requirements, and data computation procedures for evaluating the acceptability of the continuous monitoring systems.

2. Apparatus.

2.1 Calibration Gas Mixtures. Mixtures of known concentrations of pollutant gas in a diluent gas shall be prepared. The pollutant gas shall be sulfur dioxide or the appropriate oxide(s) of nitrogen specified by paragraph 6 and within subparts. For sulfur dioxide gas mixtures, the diluent gas may be air or nitrogen. For nitric oxide (NO) gas mixtures, the diluent gas shall be oxygen-free (<10 ppm) nitrogen, and for nitrogen dioxide (NO_2) gas mixtures the diluent gas shall be air. Concentrations of approximately 50 percent and 90 percent of span are required. The 90 percent gas mixture is used to set and to check the span and is referred to as the span gas.

2.2 Zero Gas. A gas certified by the manufacturer to contain less than 1 ppm of the pollutant gas or ambient air may be used.

2.3 Equipment for measurement of the pollutant gas concentration using the reference method specified in the applicable standard.

2.4 Data Recorder. Analog chart recorder or other suitable device with input voltage range compatible with analyzer system output. The resolution of the recorder's data output shall be sufficient to allow completion of the test procedures within this specification.

2.5 Continuous monitoring system for SO_2 or NO_x pollutants as applicable.

3. Definitions.

3.1 Continuous Monitoring System. The total equipment required for the determination of a pollutant gas concentration in a source effluent. Continuous monitoring systems consist of major subsystems as follows:

3.1.1 Sampling Interface. That portion of an extractive continuous monitoring system that performs one or more of the following operations: acquisition, transportation, and conditioning of a sample fo the source effluent or that portion of an in-situ continuous monitoring system that protects the analyzer from the effluent.

3.1.2 Analyzer. That portion of the continuous monitoring system which senses the pollutant gas and generates a signal output that is a function of the pollutant concentration.

3.1.3 Data Recorder. That portion of the continuous monitoring system that provides a permanent record of the output signal in terms of concentration units.

3.2 Span. The value of pollutant concentration at which the continuous monitoring system is set to produce the maximum data display output. The span shall be set at the concentration specified in each applicable subpart.

3.3 Accuracy (Relative). The degree of correctness with which the continuous monitoring system yields the value of gas concentration of a sample relative to the value given by a defined reference method. This accuracy is expressed in terms of error, which is the difference between the paired concentration measurements expressed as a percentage of the mean reference value.

3.4 Calibration Error. The difference between the pollutant concentration indicated by the continuous monitoring system and the known concentration of the test gas mixture.

3.5 Zero Drift. The change in the continuous monitoring system output over a stated period of time of normal continuous operation when the pollutant concentration at the time for the measurements is zero.

3.6 Calibration Drift. The change in the continuous monitoring system output over a stated time period of normal continuous operations when the pollutant concentration at the time of the measurements is the same known upscale value.

3.7 Response Time. The time interval from a step change in pollutant concentration at the input to the continuous monitoring system to the time at which 95 percent of the corresponding final value is reached as displayed on the continuous monitoring system data recorder.

3.8 Operational Period. A minimum period of time over which a measurement system is expected to operate within certain performance specifications without unscheduled maintenance, repair, or adjustment.

3.9 Stratification. A condition identified by a difference in excess of 10 percent between the average concentration in the duct or stack and the concentration at any point more than 1.0 meter from the duct or stack wall.

4. Installation Specifications. Pollutant continuous monitoring systems (SO_2 and NO_x) shall be installed at a sampling location where measurements can be made which are directly representative (4.1), or which can be corrected so as to be representative (4.2) of the total emissions from the affected facility. Conformance with this requirement shall be accomplished as follows:

4.1 Effluent gases may be assumed to be nonstratified if a sampling location eight or more stack diameters (equivalent diameters) downstream of any air in-leakage is selected. This assumption and data correction procedures under paragraph 4.2.1 may not be applied to sampling locations upstream of an air preheater in a stream generating facility under Subpart D of this part. For sampling locations where effluent gases are either demonstrated (4.3) or may be assumed to be nonstratified (eight diameters), a point (extractive systems) or path (in-situ systems) of average concentration may be monitored.

4.2 For sampling locations where effluent gases cannot be assumed to be nonstratified (less than eight diameters) or have been shown under paragraph 4.3 to be stratified, results obtained must be consistently representative (e.g. a point of average concentration may shift with load changes) or the data generated by sampling at a point (extractive systems) or across a path (in-situ systems) must be corrected (4.2.1 and 4.2.2) so as to be representative of the total emissions from the affected facility. Conformance with this requirement may be accomplished in either of the following ways:

4.2.1 Installation of a diluent continuous monitoring system (O_2 or CO_2 as applicable) in accordance with the procedures under paragraph 4.2 of Performance Specification 3 of this appendix. If the pollutant and diluent monitoring systems are not of the same type (both extractive or both in-situ), the extractive system must use a multipoint probe.

4.2.2 Installation of extractive pollutant monitoring systems using multipoint sampling probes or in-situ pollutant monitoring systems that sample or view emissions which are consistently representative of the total emissions for the entire cross section. The Administrator may require data to be submitted to demonstrate that the emissions sampled or viewed are consistently representative for several typical facility process operating conditions.

4.3 The owner or operator may perform a traverse to characterize any stratification of effluent gases that might exist in a stack or duct. If no stratification is present, sampling procedures under paragraph 4.1 may be applied even though the eight diameter criteria is not met.

4.4 When single point sampling probes for extractive systems are installed within the stack or duct under paragraphs 4.1 and 4.2.1, the sample may not be extracted at any point less than 1.0 meter from the stack or duct wall. Multipoint sampling probes installed under paragraph 4.2.2 may be located at any points necessary to obtain consistently representative samples.

5. Continuous Monitoring System Performance Specifications.

The continuous monitoring system shall meet the performance specifications in Table 2-1 to be considered acceptable under this method.

TABLE 2-1—PERFORMANCE SPECIFICATIONS

Parameter	Specification
1. Accuracy [1]	<20 pct of the mean value of the reference method test data.
2. Calibration error [1]	<5 pct of each (50 pct, 90 pct) calibration gas mixture value.
3. Zero drift (2 h) [1]	2 pct of span.
4. Zero drift (24 h) [1]	Do.
5. Calibration drift (2 h) [1]	Do.
6. Calibration drift (24 h) [1]	2.5 pct of span.
7. Response time	15 min maximum.
8. Operational period	168 h minimum.

[1] Expressed as sum of absolute mean value plus 95 pct confidence interval of a series of tests.

6. Performance Specification Test Procedures. The following test procedures shall be used to determine conformance with the requirements of paragraph 5. For NO$_x$ analyzers that oxidize nitric oxide (NO) to nitrogen dioxide (NO$_2$), the response time test under paragraph 6.3 of this method shall be performed using nitric oxide (NO) span gas. Other tests for NO$_x$ continuous monitoring systems under paragraphs 6.1 and 6.2 and all tests for sulfur dioxide systems shall be performed using the pollutant span gas specified by each subpart.

6.1 Calibration Error Test Procedure. Set up and calibrate the complete continuous monitoring system according to the manufacturer's written instructions. This may be accomplished either in the laboratory or in the field.

6.1.1 Calibration Gas Analyses. Triplicate analyses of the gas mixtures shall be performed within two weeks prior to use using Reference Methods 6 for SO$_2$ and 7 for NO$_x$. Analyze each calibration gas mixture (50%, 90%) and record the results on the example sheet shown in Figure 2-1. Each sample test result must be within 20 percent of the averaged result or the tests shall be repeated. This step may be omitted for non-extractive monitors where dynamic calibration gas mixtures are not used (6.1.2).

6.1.2 Calibration Error Test Procedure. Make a total of 15 nonconsecutive measurements by alternately using zero gas and each calibration gas mixture concentration (e.g., 0%, 50%, 0%, 90%, 50%, 90%, 50%, 0%, etc.). For nonextractive continuous monitoring systems, this test procedure may be performed by using two or more calibration gas cells whose concentrations are certified by the manufacturer to be functionally equivalent to these gas concentrations. Convert the continuous monitoring system output readings to ppm and record the results on the example sheet shown in Figure 2-2.

6.2 Field Test for Accuracy (Relative), Zero Drift, and Calibration Drift. Install and operate the continuous monitoring system in accordance with the manufacturer's written instructions and drawings as follows:

6.2.1 Conditioning Period. Offset the zero setting at least 10 percent of the span so that negative zero drift can be quantified. Operate the system for an initial 168-hour conditioning period in normal operating manner.

6.2.2 Operational Test Period. Operate the continuous monitoring system for an additional 168-hour period retaining the zero offset. The system shall monitor the source effluent at all times except when being zeroed, calibrated, or backpurged.

6.2.2.1 Field Test for Accuracy (Relative). For continuous monitoring systems employing extractive sampling, the probe tip for the continuous monitoring system and the probe tip for the Reference Method sampling train should be placed at adjacent locations in the duct. For NO$_x$ continuous monitoring systems, make 27 NO_x concentration measurements, divided into nine sets, using the applicable reference method. No more than one set of tests, consisting of three individual measurements, shall be performed in any one hour. All individual measurements of each set shall be performed concurrently, or within a three-minute interval and the results averaged. For SO_2 continuous monitoring systems, make nine SO_2 concentration measurements using the applicable reference method. No more than one measurement shall be performed in any one hour. Record the reference method test data and the continuous monitoring system concentrations on the example data sheet shown in Figure 2-3.

6.2.2.2 Field Test for Zero Drift and Calibration Drift. For extractive systems, determine the values given by zero and span gas pollutant concentrations at two-hour intervals until 15 sets of data are obtained. For nonextractive measurement systems, the zero value may be determined by mechanically producing a zero condition that provides a system check of the analyzer internal mirrors and all electronic circuitry including the radiation source and detector assembly or by inserting three or more calibration gas cells and computing the zero point from the upscale measurements. If this latter technique is used, a graph(s) must be retained by the owner or operator for each measurement system that shows the relationship between the upscale measurements and the zero point. The span of the system shall be checked by using a calibration gas cell certified by the manufacturer to be functionally equivalent to 50 percent of span concentration. Record the zero and span measurements (or the computed zero drift) on the example data sheet shown in Figure 2-4. The two-hour periods over which measurements are conducted need not be consecutive but may not overlap. All measurements required under this paragraph may be conducted concurrent with tests under paragraph 6.2.2.1.

6.2.2.3 Adjustments. Zero and calibration corrections and adjustments are allowed only at 24-hour intervals or at such shorter intervals as the manufacturer's written instructions specify. Automatic corrections made by the measurement system without operator intervention or initiation are allowable at any time. During the entire 168-hour operational test period, record on the example sheet shown in Figure 2-5 the values given by zero and span gas pollutant concentrations before and after adjustment at 24-hour intervals.

6.3 Field Test for Response Time.

6.3.1 Scope of Test. Use the entire continuous monitoring system as installed, including sample transport lines if used. Flow rates, line diameters, pumping rates, pressures (do not allow the pressurized calibration gas to change the normal operating pressure in the sample line), etc., shall be at the nominal values for normal operation as specified in the manufacturer's written instructions. If the analyzer is used to sample more than one pollutant source (stack), repeat this test for each sampling point.

6.3.2 Response Time Test Procedure. Introduce zero gas into the continuous monitoring system sampling interface or as close to the sampling interface as possible. When the system output reading has stabilized, switch quickly to a known concentration of pollutant gas. Record the time from concentration switching to 95 percent of final stable response. For non-extractive monitors, the highest available calibration gas concentration shall be switched into and out of the sample path and response times recorded. Perform this test sequence three (3) times. Record the results of each test on the example sheet shown in Figure 2-6.

7. Calculations, Data Analysis and Reporting.

7.1 Procedure for determination of mean values and confidence intervals.

7.1.1 The mean value of a data set is calculated according to equation 2-1.

$$\bar{x}=\frac{1}{n}\sum_{i=1}^{n}x_i \qquad \text{Equation 2-1}$$

where:

x_i=absolute value of the measurements,
Σ=sum of the individual values,
$\bar{x}$=mean value, and
n=number of data points.

7.1.2 The 95 percent confidence interval (two-sided) is calculated according to equation 2-2:

$$\text{C.I.}_{95}=\frac{t_{.975}}{n\sqrt{n-1}}\sqrt{n(\sum x_i^2)-(\sum x_i)^2} \qquad \text{Equation 2-2}$$

where:

Σx_i=sum of all data points, $t_{.975}=t_1-\alpha/2$, and
C.I._{95}=95 percent confidence interval estimate of the average mean value.

VALUES FOR $t_{.975}$

n	$t_{.975}$
2	12.706
3	4.303
4	3.182
5	2.776
6	2.571
7	2.447
8	2.365
9	2.306
10	2.262
11	2.228
12	2.201
13	2.179
14	2.160
15	2.145
16	2.131

The values in this table are already corrected for n-1 degrees of freedom. Use n equal to the number of samples as data points.

7.2 Data Analysis and Reporting.

7.2.1 Accuracy (Relative). For each of the nine reference method test points, determine the average pollutant concentration reported by the continuous monitoring system. These average concentrations shall be determined from the continuous monitoring system data recorded under 7.2.2 by integrating or averaging the pollutant concentrations over each of the time intervals concurrent with each reference method testing period. Before proceeding to the next step, determine the basis (wet or dry) of the continuous monitoring system data and reference method test data concentrations. If the bases are not consistent, apply a moisture correction to either reference method concentrations or the continuous monitoring system concentrations as appropriate. Determine the correction factor by moisture tests concurrent with the reference method testing periods. Report the moisture test method and the correction procedure employed. For each of the nine test runs determine the difference for each test run by subtracting the respective reference method test concentrations (use average of each set of three measurements for NO_x) from the continuous monitoring system integrated or averaged concentrations. Using these data, compute the mean difference and the 95 percent confidence interval of the differences (equations 2-1 and 2-2). Accuracy is reported as the sum of the absolute value of the mean difference and the 95 percent confidence interval of the differences expressed as a percentage of the mean reference method value. Use the example sheet shown in Figure 2-3.

7.2.2 Calibration Error. Using the data from paragraph 6.1, subtract the measured pollutant concentration determined under paragraph 6.1.1 (Figure 2-1) from the value shown by the continuous monitoring system for each of the five readings at each concentration measured under 6.1.2 (Figure 2-2). Calculate the mean of these difference values and the 95 percent confidence intervals according to equations 2-1 and 2-2. Report the calibration error (the sum of the absolute value of the mean difference and the 95 percent confidence interval) as a percentage of each respective calibration gas concentration. Use example sheet shown in Figure 2-2.

7.2.3 Zero Drift (2-hour). Using the zero concentration values measured each 2-hours during the field test, calculate the differences between consecutive 2-hour readings expressed in ppm. Calculate the mean difference and the confidence interval using equations 2-1 and 2-2. Report the zero drift as the sum of the absolute mean value and the confidence interval as a percentage of span. Use example sheet shown in Figure 2-4.

7.2.4 Zero Drift (24-hour). Using the zero concentration values measured every 24 hours during the field test, calculate the differences between the zero point after zero adjustment and the zero value 24 hours later just prior to zero adjustment. Calculate the mean value of these points and the confidence interval using equations 2-1 and 2-2. Report the zero drift (the sum of the absolute mean and confidence interval) as a percentage of span. Use example sheet shown in Figure 2-5.

7.2.5 Calibration Drift (2-hour). Using the calibration values obtained at 2-hour intervals during the field test, calculate the differences between consecutive 2-hour readings expressed as ppm. These values should be corrected for the corresponding zero drift during that 2-hour period. Calculate the mean and confidence interval of these corrected difference values using equations 2-1 and 2-2. Do not use the differences between non-consecutive readings. Report the calibration drift as the sum of the absolute mean and confidence interval as a percentage of span. Use the example sheet shown in Figure 2-4.

7.2.6 Calibration Drift (24-hour). Using the calibration values measured every 24 hours during the field test, calculate the differences between the calibration concentration reading after zero and calibration adjustment, and the calibration concentration reading 24 hours later after zero adjustment but before calibration adjustment. Calculate the mean value of these differences and the confidence interval using equations 2-1 and 2-2. Report the calibration drift (the sum of the absolute mean and confidence interval) as a percentage of span. Use the example sheet shown in Figure 2-5.

7.2.7 Response Time. Using the charts from paragraph 6.3, calculate the time interval from concentration switching to 95 percent to the final stable value for all upscale and downscale tests. Report the mean of the three upscale test times and the mean of the three downscale test times. The two average times should not differ by more than 15 percent of the slower time. Report the slower time as the system response time. Use the example sheet shown in Figure 2-6.

7.2.8 Operational Test Period. During the 168-hour performance and operational test period, the continuous monitoring system shall not require any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as required in the operation and maintenance manuals as routine and expected during a 1-week period. If the continuous monitoring system operates within the specified performance parameters and does not require corrective maintenance, repair, replacement or adjustment other than as specified above during the 168-hour test period, the operational period will be successfully concluded. Failure of the continuous monitoring system to meet this requirement shall call for a repetition of the 168-hour test period. Portions of the test which were satisfactorily completed need not be repeated. Failure to meet any performance specifications shall call for a repetition of the 1-week performance test period and that portion of the testing which is related to the failed specification. All maintenance and adjustments required shall be recorded. Output readings shall be recorded before and after all adjustments.

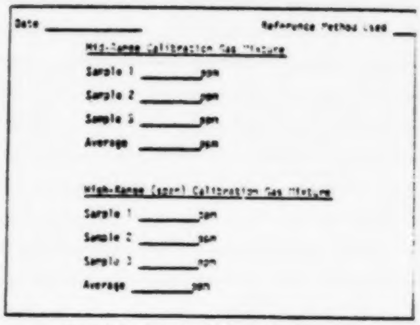
8. References.

8.1 "Monitoring Instrumentation for the Measurement of Sulfur Dioxide in Stationary Source Emissions," Environmental Protection Agency, Research Triangle Park, N.C., February 1973.

8.2 "Instrumentation for the Determination of Nitrogen Oxides Content of Stationary Source Emissions," Environmental Protection Agency, Research Triangle Park, N.C., Volume 1, APTD-0847, October 1971; Volume 2, APTD-0942, January 1972.

8.3 "Experimental Statistics," Department of Commerce, Handbook 91, 1963, pp. 3-31, paragraphs 3-3.1.4.

8.4 "Performance Specifications for Stationary Source Monitoring Systems for Gases and Visible Emissions," Environmental Protection Agency, Research Triangle Park, N.C., EPA-650/2-74-013, January 1974.



Date ________ Reference Method Used ____

Mid-Range Calibration Gas Mixture

Sample 1 ________ppm

Sample 2 ________ppm

Sample 3 ________ppm

Average ________ppm

High-Range (span) Calibration Gas Mixture

Sample 1 ________ppm

Sample 2 ________ppm

Sample 3 ________ppm

Average ________ppm

Figure 2-1. Analysis of Calibration Gas Mixtures

Calibration Gas Mixture Data (From Figure 2-1)

Mid (50%) _____ppm High (90%) _____ppm

Run #	Calibration Gas Concentration, ppm	Measurement System Reading, ppm	Differences,[1] ppm
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			

	Mid	High
Mean difference	_____	_____
Confidence interval	± _____	± _____
Calibration error = $\frac{\text{Mean Difference[2]} + \text{C.I.}}{\text{Average Calibration Gas Concentration}} \times 100$	_____%	_____%

---

[1] Calibration gas concentration - measurement system reading

[2] Absolute value

Figure 2-2. Calibration Error Determination

		Reference Method Samples								
Test No.	Date and Time	SO_2 Sample 1 (ppm)	NO_x Sample 1 (ppm)	NO_x Sample 2 (ppm)	NO_x Sample 3 (ppm)	NO_x Sample Average (ppm)	Analyzer 1-Hour Average (ppm)*		Difference (ppm)	
							SO_2	NO_x	SO_2	NO_x
1										
2										
3										
4										
5										
6										
7										
8										
9										

Mean reference method test value (SO_2) ________

Mean reference method test value (NO_x) ________

Mean of the differences ________

95% Confidence intervals = ± ________ ppm (SO_2) = ± ________ ppm (NO_x)

$$\text{Accuracies} = \frac{\text{Mean of the differences} + \text{95\% confidence interval}}{\text{Mean reference method value}} \times 100 = \_\_\_\_\% (SO_2) = \_\_\_\_\% (NO_x)$$

* Explain and report method used to determine integrated averages

Figure 2-3. Accuracy Determination (SO_2 and NO_x)

Data Set No.	Time Begin	Time End	Date	Zero Reading	Zero Drift (ΔZero)	Span Reading	Span Drift (ΔSpan)	Calibration Drift (Span- Zero)
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								

Zero Drift = [Mean Zero Drift* ________ + CI (Zero) ________] ÷ [Span] x 100 = ________.

Calibration Drift = [Mean Span Drift* ________ + CI (Span) ________] ÷ [Span] x 100 = ________.

*Absolute Value.

Figure 2-4. Zero and Calibration Drift (2 Hour)

Date and Time	Zero Reading	Zero Drift (ΔZero)	Span Reading (After zero adjustment)	Calibration Drift (ΔSpan)

Zero Drift = [Mean Zero Drift* ________ + C.I. (Zero) ________]
÷ [Instrument Span] x 100 = ________.

Calibration Drift = [Mean Span Drift* ________ + C.I. (Span) ________]
÷ [Instrument Span] x 100 = ________.

* Absolute value

Figure 2-5. Zero and Calibration Drift (24-hour)

Date of Test ____________

Span Gas Concentration ________ppm

Analyzer Span Setting ________ppm

Upscale
1 ________seconds
2 ________seconds
3 ________seconds

Average upscale response ________seconds

Downscale
1 ________seconds
2 ________seconds
3 ________seconds

Average downscale response ______seconds

System average response time (slower time) = ______seconds.

$$\%\text{deviation from slower system average response} = \left[\frac{\text{average upscale minus average downscale}}{\text{slower time}}\right] \times 100\% = \_\_\_\_.$$

Figure 2-6. Response Time

APPENDIX C

GLOSSARY OF TERMS

Abbreviations	Definitions
°Be	Degree Baume
°C	Temperature, degrees Centigrade
cc	Cubic centimeter degree Fahrenheit
ft	Feet
ft^2	Square feet
ft^3, cf	Cubic feet
°F	Temperature, degree Fahrenheit
lbs	Pounds
Mscfm	Thousands of standard dubic feet per minute
ppm	Parts per million by volume
psig	Pounds per square inch gauge
psia	Pounds per square inch absolute
scfm	Cubic feet per minute measured at standard conditions (70°F) and 760 mm (29.22") Hg
SCFH	Standard cubic feet per hour
sp.gr.	Specific gravity--compared to water at 60°F

CHEMICAL SYMBOLS

CH_4	Methane
CO_2	Carbon dioxide
H_2O	Water
HNO_3	Nitric acid
NH_3	Ammonia
N_2	Nitrogen
N_2O	Nitrous oxide (laughing gas)
NO	Nitric oxide
NO_x	Total nitrogen oxides in a mixture
NO_2	Nitrogen dioxide
N_2O_4	Nitrogen textroxide
O_2	Oxygen

Words	Definitions
Absorber	A stainless steel tower with bubble cap plates.
Baume (^{o}Be)	Acid strength is determined by use of a floating instrument (hydrometer) calibrated to read ^{o}Be and by a conversion chart. The Baume can also be calculated if the specific gravity of the acid at $60^{o}F$ is known: $^{o}Be = 145 -- \left(\frac{145}{sp.gr.}\right)$
Catalyst	The platinum-rhodium woven wire gauze on which the ammonia is oxidized to nitric oxide and water.
Catalytic reduction system	A device for reducing the emissions of nitrogen oxides to the atmosphere by passing the gas over a catalyst in the presence of a reducing fuel such as natural gas, hydrogen or ammonia.
Converter	The chamber in which the ammonia is converted to nitric oxide and water by reacting it with air over a platinum-rhodium catalyst.
Effluent	Waste gas stream that enters the atmosphere from the process.
Emission	Any gas stream emitted to the atmosphere.
Nitric acid (weak)	Thirty to seventy percent nitric acid.
Nitrogen oxides	A general term pertaining to a mixture of nitric oxide (NO) and nitrogen dioxide (NO_2).
Tail gas	The gas leaving the nitric acid absorber.

*U.S. GOVERNMENT PRINTING OFFICE: 1985--537-002/21,507